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A.S.T.M. STANDARDS

Including

TENTATIVE STANDARDS

(A Triennial Publication)

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PART II

Nonmetallic Materials—Constructional

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FOREWORD

The Book of A.S.T.M. Standards, of which this is the 1944 edition, is normally a triennial[1] publication, with Supplements issued in the intervening years. It contains the formally adopted A.S.T.M. Standards and the A.S.T.M. Tentative Standards as well. The present edition, in view of the War Emergency, also includes Emergency Standards and Emergency Alternate Provisions in A.S.T.M. Standards issued under an emergency procedure in the interest of expediting procurement or conservation of materials.

The Book is issued in three parts:

Part I, Metals.—Ferrous and non-ferrous metals (except methods of chemical analysis), general testing methods. The methods of chemical analysis of metals appear in a separate publication bearing that title.

Part II, Nonmetallic Materials—Constructional.—Cementitious materials, concrete and aggregates, masonry building units, pipe and tile, thermal insulating materials, refractories, glass, wood and wood preservatives, paints, varnishes and lacquers, naval stores, road materials, soils, waterproofing and roofing materials, fire tests, general testing methods, thermometers.

Part III, Nonmetallic Materials—General.—Coal and coke, petroleum products, paper, electrical insulating materials, plastics, rubber, textiles, soaps and detergents, naval stores, water, general testing methods, thermometers.

In these three parts are included the 1235 A.S.T.M. Standards and Tentative Standards in effect at the time of publication, exclusive of the 22 A.S.T.M. Methods of Chemical Analysis of Metals which appear in a separate publication bearing that title.

The annual Supplements issued to each part in the succeeding years will contain the newly adopted or revised Standards and new or revised Tentative Standards. These Supplements should be consulted for changes in status and for current revisions of Standards. The combined Index to Standards and Tentative Standards, issued annually, will also be helpful in this connection and in locating any desired standard.

Standards appear in the front of each part, and comprise those specifications and methods of test that have been formally adopted by the Society, requiring a letter ballot approval by the entire membership.

Tentative Standards, appearing in the back of each part, are published as tentative by the Society on the recommendation of the committee concerned, as representing the latest thoughts and practices, prior to adoption as standard.

Emergency Standards and *Emergency Alternate Provisions* are intended for use during the National Emergency where they may be considered as suitable for the specific application or use desired or as a permissible alternate for existing specifications.

The Emergency Standards and Emergency Alternate Provisions applying to the standards in this part of the Book of Standards appear in a separate section on pink colored stock in the middle of the book. For convenience, the Emergency Alternate Provisions may be cut out and attached to the standards to which they apply.

In accordance with Society regulations, standards that have been published for a period of six years or more without revision have been reviewed by the responsible standing committees prior to the appearance of the Book and have been reapproved in their present form.

The appearance of this Book is a tribute to the sincere efforts of the standing committees responsible for the development and review of the standards, and of the many individuals serving on the standing committees. (See list of committees appearing on page 1611.) The Society's time-tested standardization procedure provides for adequate representation on technical committees of producers and consumers and those having a general interest in the material or subject in question. Opportunity is provided during the period of promulgation for full consideration by all interested parties through publication as tentative before adoption as standard. As developments and advances take place, requirements are suitably revised.

GUIDE TO THE USE OF THE BOOK OF STANDARDS

THE standards in this Book are assembled in a sequence determined by the specific materials or products to which they apply. The Table of Contents in each part is presented in duplicate—one a list of standards in the order in which they appear, the other a list of the standards in numeric sequence of their serial designations. In addition, a separate listing (printed on green colored stock) is given of the tentative standards, just preceding that portion of the Book (see page opposite 1202). The Tentative Revisions of Standards appear in a separate section beginning on page 1601.

Emergency Standards and Emergency Alternate Provisions applying to the standards in this book appear in the pink colored insert section beginning on page 1172.

A subject index of the standards and tentative standards in this part appears at the back of this volume (see page 1613). This index should be the most convenient means of locating any standard appearing in the volume, especially if only the general subject matter covered is known.

A separate combined Index, covering all standards and tentative standards in Parts I, II, and III of the Book of A.S.T.M. Standards, in the annual Supplements thereto, and in the book of A.S.T.M. Methods of Chemical Analysis of Metals, is also issued annually, which should be of convenience to all users of the Book of Standards. The combined Index will be revised annually to incorporate the latest references to the standards, whether they appear in the Book of Standards or in the 1945 or 1946 Supplements. The combined Index will be particularly helpful after one of the Supplements has been issued.

Comments or suggestions intended to improve the utility of the Book of Standards will be welcome.

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(For standards covering other nonmetallic materials, see Part III)

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Emergency Standards appear in a separate section beginning on p. 1172, and are printed on colored stock. They are identified by the letters ES in their serial designation. A letter following the number indicates revisions made subsequent to the original date of issue, for example, a for the first revision, b for the second, etc.

Emergency Alternate Provisions also appear in a separate section beginning on p. 1172 and are identified by the letters EA followed by the permanent designation of the standard to which they apply.

In the following listing, the standards are grouped according to materials to which they apply. Listings of Tentative Standards appear in italics.

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For a list of Emergency Standards and Emergency Alternate Provisions, see p. 1172.

The Tentative Standards are listed separately in the green insert opposite p. 1202.

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[1] This is in effect a tentative revision of, and, when adopted, is to replace Standard Specifications C 91 – 40.
[2] This is in effect a tentative revision of, and, when adopted, is to be added to Standard Methods C 114 – 44.

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† Approved as **American Standard** by the American Standards Association.
‡ A "tentative revision" of this standard appears in the Tentative Revisions of Standards section at the back of this book, see p. 1601.
[3] This is in effect a tentative revision of, and, when adopted, is to be added to Standard Definitions C 51 – 44.

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† Approved as **American Standard** by the American Standards Association.
‡ A "tentative revision" of this standard appears in the Tentative Revisions of Standards section at the back of this book, see p. 1601.
[4] This is in effect a tentative revision of, and, when adopted, is to be added to Standard Definitions C 11 – 41.

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† Approved as **American Standard** by the American Standards Association.
[5] These specifications were formerly published as Emergency Specifications ES – 8.
[6] These specifications were formerly published as Emergency Specifications ES – 9.
[7] These specifications were formerly published as Emergency Specifications ES – 10.
[8] These specifications were formerly published as Emergency Specifications ES – 11.
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‡ A "tentative revision" of this standard appears in the Tentative Revisions of Standards section at the back of this book, see p. 1601.

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‡ A "tentative revision" of this standard appears in the Tentative Revisions of Standards section at the back of this book, see p. 1601.

† Approved as **American Standard** by the American Standards Association.

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† Approved as **American Standard** by the American Standards Association.

[10] This is in effect a tentative revision of, and, when adopted, is to be added to Standard Definitions C 58 – 28.

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[11] This is in effect a tentative revision of, and, when adopted, is to be added to Standard Methods D 29 – 40.

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† Approved as **American Standard** by the American Standards Association.

[13] This is in effect a tentative revision of, and, when adopted, is to be added to Standard Specifications E 1 – 44.

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† Approved as **American Standard** by the American Standards Association.
‡ A "tentative revision" of this standard appears in the Tentative Revisions of Standards section at the back of this book, see p. 1601.
[1] This is in effect a tentative revision of, and, when adopted, is to be added to Standard Definitions C 11 – 41.

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‡ A "tentative revision" of this standard appears in the Tentative Revisions of Standards section at the back of this book, see p. 1601.

[2] This is in effect a tentative revision of, and, when adopted, is to be added to Standard Definitions C 51 – 44.

[3] This is in effect a tentative revision of, and, when adopted, is to be added to Standard Definitions C 58 – 28.

[4] This is in effect a tentative revision of, and, when adopted, is to replace Standard Specifications C 91 – 40.

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‡ A "tentative revision" of this standard appears in the Tentative Revisions of Standards section at the back of this book, see p. 1601.
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[6] This is in effect a tentative revision of, and, when adopted, is to be added to Standard Methods D 29 – 40.

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† This is in effect a tentative revision of, and, when adopted, is to be added to Standard Methods D 268 – 44.

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EMERGENCY STANDARDS

A List in Numeric Sequence of the Emergency Standards applying to nonmetallic materials—constructional, appears in the pink section on page 1172.

EMERGENCY ALTERNATE PROVISIONS

A List in Numeric Sequence of the Emergency Alternate Provisions applying to nonmetallic materials—constructional, appears in the pink section on page 1172.

* Emergency Alternate Provisions applying to this standard have been issued and appear in the pink section of this book, see p. 1172.
† Approved as **American Standard** by the American Standards Association.
[a] This is in effect a tentative revision of, and, when adopted, is to be added to Standard Specifications E 1 – 44.

Standard Specifications for

PORTLAND CEMENT[1]

A.S.T.M. Designation: C 150 – 44

ADOPTED, 1941; REVISED, 1942, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 150; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover five types of portland cement, as follows:

Type I.—For use in general concrete construction when the special properties specified for types II, III, IV, and V are not required.

Type II.—For use in general concrete construction exposed to moderate sulfate action, or where moderate heat of hydration is required.

Type III.—For use when high early strength is required.

Type IV.—For use when a low heat of hydration is required (Note).

Type V.—For use when high sulfate resistance is required (Note).

NOTE.—Attention is called to the fact that cements conforming to the requirements for type IV and type V are not usually carried in stock. In advance of specifying their use, purchasers or their representatives should determine whether these types of cement are, or can be made available.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-1 on Cement.

[2] Prior to adoption as standard, these specifications were published as tentative from 1940 to 1941, being revised in 1941.

These specifications replace the former Standard Specifications for Portland Cement (C 9 – 38), and High-Early-Strength Portland Cement (C 74 – 39), which were accordingly discontinued in September, 1941.

Basis of Purchase

2. The purchaser should specify the type or types desired. When no type is specified, the requirements of type I shall govern.

Definition

3. For the purpose of these specifications, portland cement is the product obtained by pulverizing clinker consisting essentially of hydraulic calcium silicates, to which no additions have been made subsequent to calcination other than water and/or untreated calcium sulfate, except that additions not to exceed 1.0 per cent of other materials may be interground with the clinker at the option of the manufacturer, provided such materials in the amounts indicated have been shown to be not harmful by tests carried out or reviewed by Committee C-1 on Cement[3] (Note).

NOTE.—Tests to determine whether a proposed addition is not harmful will be carried out or reviewed by Committee C-1 on Cement, for

[3] The committee has declared as not harmful the inclusion of the material known commercially as TDA (composed of triethanolamine and highly purified soluble calcium salts of modified lignin sulfonic acids), manufactured by the Dewey and Almy Chemical Co., when added in an amount not exceeding 0.043 per cent by weight of the cement, except that in type III cement a maximum of 0.08 per cent by weight may be used.

those making requests. In the year following that in which an addition has been declared not harmful by the Committee, the name of the addition and the amount permitted shall appear as an addendum to Section 3 in a revision of the specifications.

Chemical Requirements

4. Portland cement of each of the five types shown in Section 1 shall conform to the respective chemical requirements prescribed in Table I.

TABLE I.—CHEMICAL REQUIREMENTS.

	Type I	Type II	Type III	Type IV[a]	Type V[a]
Silicon dioxide (SiO_2), min., per cent	...	21.0	...	...	24.0
Aluminum oxide (Al_2O_3), max., per cent	...	6.0	...	...	4.0
Ferric oxide (Fe_2O_3), max., per cent	...	6.0	...	6.5	4.0
Magnesium oxide (MgO), max., per cent	5.0	5.0	5.0	5.0	4.0
Sulfur trioxide (SO_3), max., per cent	2.0	2.0	2.5	2.0	2.0
Loss on ignition, max., per cent	3.0	3.0	3.0	2.3	3.0
Insoluble residue, max., per cent	0.75	0.75	0.75	0.75	0.75
Ratio of Al_2O_3 to Fe_2O_3	...	0.7 to 2.0	...	...	0.7 to 2.0
Tricalcium silicate ($3CaO \cdot SiO_2$),[b] max., per cent	...	50	...	35	...
Dicalcium silicate ($2CaO \cdot SiO_2$),[b] min., per cent	...	...	...	40	...
Tricalcium aluminate ($3CaO \cdot Al_2O_3$),[b] max., per cent	...	8	15	7	5

[a] See Note, Section 1.

[b] The expressing of chemical limitations by means of calculated assumed compounds does not necessarily mean that the oxides are actually or entirely present as such compounds.

The percentages of tricalcium silicate, dicalcium silicate, and tricalcium aluminate shall be calculated from the chemical analysis as follows:

Tricalcium silicate =
(4.07 × per cent CaO) − (7.60 × per cent SiO_2)
− (6.72 × per cent Al_2O_3) − (1.43 × per cent Fe_2O_3) − (2.85 × per cent SO_3)

Dicalcium silicate =
(2.87 × per cent SiO_2) − (0.754 × per cent $3CaO \cdot SiO_2$)

Tricalcium aluminate =
(2.65 × per cent Al_2O_3) − (1.69 × per cent Fe_2O_3)

Oxide determinations calculated to the nearest 0.1 per cent shall be used in the calculations. Compound percentages shall be calculated to the nearest 0.1 per cent and reported to the nearest 1 per cent.

Physical Requirements

5. Portland cement of each of the five types shown in Section 1 shall conform to the respective physical requirements prescribed in Table II.

Packaging and Marking

6. When cement is delivered in packages, the name and brand of the manufacturer and the type under these specifications shall be plainly indicated thereon, except that in the case of type I the type need not be indicated. Similar information shall be provided in the shipping advices accompanying the shipment of packaged or bulk cement. A bag shall contain 94 lb. net. A barrel shall consist of 376 lb. net. All packages shall be in good condition at the time of inspection.

Storage

7. The cement shall be stored in such a manner as to permit easy access for proper inspection and identification of each shipment, and in a suitable weather-tight building that will protect the cement from dampness and minimize warehouse set.

Inspection

8. Every facility shall be provided the purchaser for careful sampling and inspection, either at the mill or at the site of the work, as may be specified by the purchaser. The following periods from time of sampling shall be allowed for completion of testing:

1-day test	6 days
3-day test	8 days
7-day test	12 days
28-day test	33 days

Rejection

9. (*a*) The cement may be rejected if it fails to meet any of the requirements of these specifications.

(*b*) Cement remaining in bulk storage

at the mill, prior to shipment, for a period greater than 6 months after completion of the tests may be retested and may be rejected if it fails to conform to any of the requirements of these specifications.

(*c*) Packages varying more than 5 per cent from the specified weight may be rejected; and if the average weight of packages in any shipment, as shown by weighing 50 packages taken at random, is less than that specified, the entire shipment may be rejected.

TABLE II.—PHYSICAL REQUIREMENTS.

	Type I	Type II	Type III	Type IV[a]	Type V[a]
Fineness, specific surface, sq. cm. per g.:					
Average value, min.	1600	1700	...	1800	1800
Minimum value, any one sample	1500	1600	...	1700	1700
Soundness:					
Autoclave expansion, max., per cent	0.50	0.50	0.50	0.50	0.50
Time of setting (alternate methods):[b]					
Gillmore test:					
Initial set, min., not less than	60	60	60	60	60
Final set, hr., not more than	10	10	10	10	10
Vicat test:					
Initial set, min., not less than	45	45	45	45	45
Final set, hr., not more than	10	10	10	10	10
Compressive strength, psi.:[c] The compressive strength of mortar cubes, composed of 1 part cement and 2.75 parts graded standard sand, by weight, prepared and tested in accordance with Method C 109, shall be equal to or higher than the values specified for the ages indicated below:					
1 day in moist air	...	...	1250	...	...
1 day in moist air, 2 days in water	900	750	2500	...	...
1 day in moist air, 6 days in water	1800	1500	...	800	1000
1 day in moist air, 27 days in water	3000	3000	[c]	2000	2200
Tensile strength, psi.:[c] The tensile strength of mortar briquets composed of 1 part cement and 3 parts standard sand, by weight, prepared and tested in accordance with Method C 190, shall be equal to or higher than the values specified for the ages indicated below:					
1 day in moist air	...	...	275	...	...
1 day in moist air, 2 days in water	150	125	375	...	...
1 day in moist air, 6 days in water	275	250	...	175	175
1 day in moist air, 27 days in water	350	325	[c]	300	300

[a] See Note, Section 1.

[b] The purchaser should specify the type of setting time test required. In case he does not so specify, the requirement of the Gillmore test only shall govern.

[c] The purchaser should specify the type of strength test required. In case he does not so specify, the requirements of the tensile strength test only shall govern. The strength at any age shall be higher than the strength at the next preceding age. Unless otherwise specified, the compressive and tensile strength tests for types I and II cement will be made only at 3 and 7 days. If, at the option of the purchaser, a 28-day test is required on type III cement, the strength at 28 days shall be higher than at 3 days.

Methods of Testing

10. The cement shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(*a*) *Sampling.*—Standard Methods of Sampling Hydraulic Cement (A.S.T.M. Designation: C 183).[4]

(*b*) *Chemical Analysis.*—Standard Methods of Chemical Analysis of Portland Cement (A.S.T.M. Designation: C 114 - 44)[4] and Tentative Methods of Chemical Analysis of Portland Cement (A.S.T.M. Designation: C 114 - 44 T).[4]

(*c*) *Fineness.*—Standard Method of Test for Fineness of Portland Cement by the Turbidimeter (A.S.T.M. Designation: C 115).[4]

(*d*) *Autoclave Expansion.*—Standard

[4] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Method of Test for Autoclave Expansion of Portland Cement (A.S.T.M. Designation: C 151).[4]

(*e*) *Time of Setting.*—Standard Methods of Test for Time of Setting of Hydraulic Cement by the Vicat or Gillmore Needles (A.S.T.M. Designation: C 191).[4]

(*f*) *Compressive Strength.*—Standard Method of Test for Compressive Strength of Hydraulic-Cement Mortars (A.S.T.M. Designation: C 109).[4]

(*g*) *Tensile Strength.*—Standard Method of Test for Tensile Strength of Hydraulic-Cement Mortars (A.S.T.M. Designation: C 190).[4]

Standard Specifications for
NATURAL CEMENT[1]

A.S.T.M. Designation: C 10 – 37

ADOPTED, 1904; REVISED, 1908, 1909, 1937.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 10; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover natural cement.

Definition

2. Natural cement is the product obtained by finely pulverizing calcined argillaceous limestone, to which not to exceed 5 per cent of nondeleterious materials may be added subsequent to calcination. The temperature of calcination shall be no higher than is necessary to drive off carbonic acid gas.

Fineness

3. The residue on a standard No. 200 (74-micron) sieve shall not exceed 15 per cent by weight.

Soundness

4. A pat of neat cement, about 3 in. in diameter, and 0.5 in. in thickness at the center and tapering to a thin edge, shall be kept in moist air for a period of 48 hr. The pat shall then be placed in an atmosphere of steam at a temperature between 98 and 100 C. on a suitable support 1 in. above boiling water for 5 hr. and shall show no signs of distortion, cracking, checking, or disintegration upon removal from the steam chest.

Time of Setting

5. The cement shall not develop initial set in less than 10 min. when the Vicat needle is used or in less than 20 min. when the Gillmore needle is used. Final set shall be attained within 10 hr.

Tensile Strength

6. (*a*) The average tensile strength of not less than three standard mortar briquets (see Section 11) composed of one part of cement and two parts of standard sand, by weight, shall be equal to or higher than the following:

AGE AT TEST, DAYS	STORAGE OF SPECIMENS	TENSILE STRENGTH, PSI.
7	1 day in moist air, 6 days in water....	75
28	1 day in moist air, 27 days in water...	150

(*b*) The average tensile strength of standard mortar at 28 days shall be higher than the strength at 7 days.

Packages and Marking

7. The cement shall be delivered in packages as specified with the brand and

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-1 on Cement.

[2] Editorially revised in 1939.

name of the manufacturer plainly marked thereon, unless shipped in bulk. When shipped in bulk, this information shall be contained in the shipping advices accompanying the shipment. A barrel shall contain 282 lb. net. All packages shall be in good condition at the time of inspection.

Storage

8. The cement shall be stored in such a manner as to permit easy access for proper inspection and identification of each shipment, and in a suitable weather-tight building which will protect the cement from dampness.

Inspection

9. Every facility shall be provided the purchaser for careful sampling and inspection either at the mill or at the site of the work, as may be specified by the purchaser. At least 12 days from the time of sampling shall be allowed for completion of the 7-day test and at least 33 days shall be allowed for completion of the 28-day test.

Rejection

10. (*a*) The cement may be rejected if it fails to conform to any of the requirements of these specifications.

(*b*) Cement remaining in storage prior to shipment for a period greater than 6 months after completion of the tests shall be retested and shall be rejected if it fails to conform to any of the requirements of these specifications.

(*c*) Cement shall not be rejected on account of failure to conform to the fineness requirement if upon retest after drying at 100 C. for 1 hr. it meets this requirement.

(*d*) Cement failing to meet the test for soundness in steam may be accepted if it passes a retest using a new sample at any time within 28 days thereafter. The provisional acceptance of the cement at the mill shall not deprive the purchaser of the right of rejection on a retest of soundness and time of setting at the time of delivery of cement to the purchaser.

Methods of Testing[3]

11. The cement shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials, except as otherwise prescribed in these specifications:

(*a*) *Sampling.*—Standard Methods of Sampling Hydraulic Cement (A.S.T.M. Designation: C 183).[4]

(*b*) *Fineness.*—Standard Method of Test for Fineness of Hydraulic Cement by the No. 200 Sieve (A.S.T.M. Designation: C 184).[4]

(*c*) *Soundness.*—Standard Method of Test for Soundness of Hydraulic Cement Over Boiling Water (Pat Test) (A.S.T.M. Designation: C 189),[4] except that immediately after being made the pat shall be stored in moist air for 48 hr. and then placed in an atmosphere of steam at a temperature between 98 and 100 C. on a suitable support 1 in. above boiling water for 5 hr.

(*d*) *Time of Setting.*—Standard Methods of Test for Time of Setting of Hydraulic Cement by the Vicat or Gillmore Needles (A.S.T.M. Designation: C 191).[4]

(*e*) *Tensile Strength.*—Standard Method of Test for Tensile Strength of Hydraulic-Cement Mortars (A.S.T.M. Designation: C 190),[4] except that the proportions of the standard mortar shall be one part of cement to two parts of standard sand, by weight.

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[3] This section was editorially revised in 1944.

[4] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for

MASONRY CEMENT[1]

A.S.T.M. Designation: C 91 – 40

ADOPTED, 1940.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 91; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover cement to be used in conjunction with sand in the making of mortar for laying units of masonry construction.

Time of Setting

2. Neat cement paste mixed to normal consistency shall not develop initial set in less than 60 min. as determined by the Gillmore needle. Final set shall be attained within 24 hr.

Soundness

3. A pat of neat cement shall remain firm and hard, and show no signs of distortion, cracking, checking, or disintegration in the steam test for soundness.

Compressive Strength

4. The average compressive strength of not less than three 2-in. cubes, made, stored, and tested in accordance with Sections 19 to 25, shall be not less than 350 psi. at the age of 7 days. The average strength at 28 days shall be not less than 600 psi.

Water Retention

5. Cement mortar after suction for 60 sec. shall have a flow greater than 65 per cent of that immediately after mixing, when tested in accordance with the procedure described in Section 26.

Staining

6. This requirement shall apply only when the invitation for bids specifically states that the cement shall be nonstaining to limestone. Nonstaining cement shall contain not more than 0.03 per cent of water-soluble "alkali" when tested in accordance with Section 27.

NOTE.—The amount and nature of the staining material in limestones seem to vary with the stone. The alkali in any cement may, therefore, induce markedly different staining on different stone, even though the stone may have come apparently from the same source. The amount of alkali permitted by the specifications should not cause stain unless stone high in staining material has been used, or unless insufficient means have been used to prevent infiltration of water into the masonry.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-1 on Cement.

[2] Prior to adoption as standard, these specifications were published as tentative from 1932 to 1940, being revised in 1938 and 1940. Editorially revised in 1944.

A "tentative revision" of this standard appears in the Tentative Revisions of Standards section at the back of this book.

Packaging and Marking

7. The cement shall be delivered in suitable bags or barrels with the brand and name of the manufacturer and the net weight plainly marked thereon. All packages shall be in good condition at the time of inspection. Packages varying more than 5 per cent from the weight marked thereon may be rejected, and if the average weight of packages in any shipment as shown by weighing 50 packages taken at random is less than the weight marked thereon, the entire lot represented may be rejected.

Storage

8. The cement shall be stored in a suitable weathertight building that will protect it from dampness, and in such a manner as to permit safe and easy access for proper inspection and identification of each lot.

Inspection

9. Every facility shall be provided the purchaser for the necessary inspection and sampling.

Rejection

10. The cement shall be rejected if it fails to meet any of the requirements of these specifications. Cement remaining in storage prior to shipment for a period greater than 6 months after test shall be retested, and shall be rejected if it fails to meet any of the requirements of these specifications.

SAMPLING AND METHODS OF TESTING

Size and Number of Samples

11. Each sample of cement selected for purpose of tests shall weigh at least 5 lb. and shall represent not more than 300 bbl. If only one sample is taken, it shall weigh at least 10 lb.

Shipment of Samples

12. Samples shall be shipped and stored in airtight, moistureproof containers.

Temperature

13. The temperature of the room and dry materials shall be maintained at not less than 20 C. (68 F.) and not more than 27.5 C. (81.5 F.). The temperature of the mixing water, moist closet, and water in the briquet storage tank shall not vary from 21 C. (70 F.) by more than 1.7 C (3 F.).

Preparation of Samples

14. The sample of cement shall be given a preliminary sieving by being passed through a No. 20 (840-micron) sieve[3] in order to thoroughly mix the sample, break up lumps, and remove foreign materials.

Mixing

15. (*a*) *Pastes.*—A 500-g. portion of the cement shall be placed in the form of a crater on a plane, nonabsorbent plate. About one half of the necessary mixing water shall be poured into the crater, the cement mixed into the water with the finger tips until all the water is taken up by the cement, the remainder of the water added, and the paste then mixed with the fingers until all the water is absorbed. The mixing operation shall then be completed by continuously and vigorously squeezing and kneading the paste with the hands for at least 3 min. During the operation of mixing, the hands shall be protected by rubber gloves.

(*b*) *Mortars.*—The mortars shall be mixed in a nonabsorbent bowl of about 1-gal. capacity. A measured quantity

[3] Detailed requirements for these sieves are given in the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

of water shall be poured into the bowl which has previously been wiped with a damp cloth. A 500-g. portion of the cement shall then be added and stirred into the water with the fingers of one hand until all the cement is wetted. Approximately 800 g. of sand shall then be added and the stirring continued for 30 sec. The remainder of 1500 g. of sand shall then be added and the mortar mixed for 75 sec. by vigorous and continued stirring, squeezing, and kneading with one hand. The mortar shall then be allowed to stand for 60 sec. and then mixed for another 60 sec. During the operation of mixing, the hands shall be protected by rubber gloves.

Normal Consistency[4]

16. Normal consistency shall be determined by the Vicat apparatus in accordance with the Standard Method of Test for Normal Consistency of Hydraulic Cement (A.S.T.M. Designation: C 187),[5] except that the mixing of the paste shall be performed in accordance with Section 15 (*a*) of these specifications.

Time of Setting Test[4]

17. The time of setting shall be determined by the Gillmore needle method in accordance with the Standard Methods of Test for Time of Setting of Hydraulic Cement by the Vicat or Gillmore Needles (A.S.T.M. Designation: C 191),[5] except that the mixing of the paste shall be performed in accordance with Section 15 (*a*) of these specifications.

Soundness Test[4]

18. Pats of neat cement, made and tested in accordance with the Standard Method of Test for Soundness of Hydraulic Cement over Boiling Water (Pat Test) (A.S.T.M. Designation: C 189),[5] except that the pastes shall be prepared in accordance with Section 15 (*a*) of these specifications, shall remain firm and hard and show no signs of distortion, checking, cracking, or disintegration.

Compressive Strength Test

19. *Molds* (*a*).—The compression test specimens shall be 2-in. cubes. Molds for the specimens shall be tight fitting. The parts of the molds, when assembled, shall be positively held together. The molds shall be made of hard metal not attacked by the cement mortar. For new molds, the Rockwell hardness number of the metal shall be not less than B55 (the Brinell hardness number not less than 95). There shall be sufficient material in the sides of the molds to prevent spreading or warping.

(*b*) The interior faces of the molds shall be true plane surfaces with a permissible variation of 0.001 in. for new molds and 0.002 in. for molds in use.

(*c*) The distances between opposite faces of the molds shall be 2 ± 0.005 in. for new molds, or 2 ± 0.01 in. for molds in use. The height of the molds, measured separately for each cube compartment, shall be 2 in. with a permissible variation of plus 0.01 in. and minus 0.005 in. for new molds, or plus 0.01 in. and minus 0.015 in. for molds in use.

(*d*) The angle between adjacent interior faces and between interior faces and top and bottom planes of the mold shall be 90 ± 0.5 deg.

(*e*) Molds shall be oiled with a mineral oil before using.

20. *Standard Sand.*[6]—The sand used for making test specimens shall be natural silica sand from Ottawa, Ill., graded to pass a No. 20 (840-micron) sieve and retained on a No. 30 (590-micron)

[4] This section was editorially revised in 1944.

[5] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

[6] This section was added editorially in 1944.

sieve.[3] This sand shall be considered standard when not more than 15 g. are retained on the No. 20 sieve, and not more than 5 g. pass the No. 30 sieve after 5 min. of continuous sieving of a 100-g. sample in the manner specified for sieving cement in the Standard Method of Test for Fineness of Hydraulic Cement by the No. 200 Sieve (A.S.T.M. Designation: C 184).[5]

21. (*a*) *Proportions for Standard Mortar.*[4]—The mortar shall consist of 1 part of cement to 3 parts of standard sand by weight.

(*b*) *Preparation of Standard Mortar.*—The mortar shall be mixed with a measured quantity of water in accordance with the procedure described in Section 15 (*b*). The top of the flow table (Paragraph (*c*)) shall be carefully wiped dry and the flow mold placed at the center and filled with mortar. In filling the mold, the mortar shall not be rammed, but gently smoothed off level with the top of the mold by aid of a trowel and the mold then removed. Immediately, the table shall be dropped through a height of $\frac{1}{2}$ in., 25 times in 15 sec. The flow is the resulting increase in diameter of the mortar mass, expressed as a percentage of the original diameter. The mortar shall be of standard consistency when the flow is 65 to 80 per cent. Trial mortars shall be made with varying percentages of water until the standard consistency is obtained. Each trial shall be made with fresh mortar. The quantity of water shall be expressed as a percentage of the weight of the combined dry materials.

(*c*) *Flow Table.*—The flow table apparatus consists of a rigid frame with a flat circular top, so mounted on a vertical shaft that it can be raised and dropped through a fixed height of $\frac{1}{2} \pm \frac{1}{32}$ in. by means of a rotated cam. The top shall be of noncorrodible metal 10 in. in diameter and with the attached shaft shall weigh 9 lb. $\pm$ 1 oz. The frame shall be attached rigidly to a concrete pedestal, which in turn shall be attached rigidly to the floor. The concrete pedestal shall be at least 8 in. in diameter and 25 in. in height and shall weigh at least 100 lb. The mold shall be of a noncorrodible material, 4 in. in inside diameter at the base, $2\frac{3}{4}$ in. at the top, and 2 in. in height.

22. *Molding of Test Cubes.*—Immediately after mixing the standard mortar in accordance with the procedure described in Sections 15 and 21, the mortar shall be placed in the cube molds which shall rest on plane, nonabsorbent plates. The molds shall be filled heaping full without compacting. Then the mortar shall be gently worked into the molds with the thumbs, using only sufficient effort to insure uniform filling of the molds. Additional mortar shall be heaped above the molds and smoothed off with a trowel. The molds and plates shall then be turned over upon plane, nonabsorbent plates oiled with mineral oil, the first plates removed, and the operation of heaping, thumbing, and smoothing off repeated. No ramming or tamping shall be used, nor any troweling in excess of that required to smooth off the specimens.

23. *Storage of Test Cubes.*—All test specimens, immediately after molding, shall be kept in the molds on plane plates in a damp closet, maintained at a relative humidity of 90 per cent or more, for from 48 to 52 hr. in such a manner that the upper surfaces shall be exposed to the moist air. The cubes shall then be removed from the molds and placed in the damp closet for 5 days in such a manner as to allow free circulation of air around at least five faces of the specimens. At the age of 7 days the cubes for the 28-day tests shall be immersed in clean running

water in storage tanks of noncorrodible materials.

24. *Testing of Cubes* (*a*).—The testing machine for determination of the compressive strength may be of either the hydraulic or the screw-type with sufficient opening between the upper bearing surface and the lower bearing surface of the machine to permit the use of verifying apparatus. The load applied to the test specimen shall be indicated with an accuracy of plus or minus 1.0 per cent.

(*b*) The upper bearing shall be a spherically seated, hardened metal block firmly attached at the center of the upper head of the machine. The center of the sphere shall lie at the center of the surface of the block in contact with the specimen. The block shall be closely held in its spherical seat, but shall be free to turn in any direction. The diagonal or diameter of the bearing surface shall be only slightly greater than the diagonal of the face of the 2-in. cube, in order to facilitate accurate centering of the specimen. A hardened metal bearing block shall be used beneath the specimen to minimize wear of the lower plate of the machine. The bearing block surface intended for contact with the specimen should have a hardness not less than Rockwell number C60 (Brinell number 620). These surfaces shall not depart from plane surfaces by more than 0.0005 in. when the blocks are new, and shall be maintained within a permissible variation of 0.001 in.

(*c*) Testing of the cube specimens shall be carried out immediately upon their removal from the moist closet for 7-day specimens, and from storage water for all other specimens. If more than one specimen at a time is removed from the storage water for testing, these cubes shall be placed in a pan of water of sufficient depth to completely immerse each cube until its removal for testing. The load shall be applied to the faces of the cubes that were in contact with the true plane surfaces of the mold. These faces shall be checked by application of an accurate straightedge. If appreciable curvature is present, the face or faces shall be ground to a plane surface before loading, or the specimens shall be discarded.

(*d*) Loose sand grains or incrustations shall be removed from the contact faces, and the cubes shall then be carefully placed in the testing machine below the center of the upper bearing block. No cushioning or bedding materials shall be used. The loading up to 25 per cent of the expected maximum load may be applied at any convenient rate, after which the specimens shall be loaded continuously to failure at a rate or rates which shall at no time be less than 1000 nor more than 6000 psi. per min.

25. *Calculations.*—The total maximum load indicated by the testing machine shall be recorded, and the compressive strength calculated in pounds per square inch from the cross-sectional area of the cube tested. Cubes that are manifestly faulty or that give strengths differing by more than 10 per cent from the average value of all test specimens made from the same sample and tested at the same period shall not be considered in determining the compressive strength.

Water Retention Test

26. (*a*) *Apparatus.*—For the water retention test, an apparatus essentially the same as that shown in Fig. 1 shall be used. This apparatus consists of a water aspirator controlled by a mercury column relief and connected by way of a three-way stopcock to a funnel upon which rests a perforated dish. A mercury manometer, connected as shown in

Fig. 1, indicates the vacuum. A rubber gasket shall be sealed to the top of the funnel and shall be kept wet during a test to insure a seal between the funnel and dish. The perforated dish shall be made of nonabsorbent material. Hardened filter paper equivalent to Carl

diately after making the flow test, the mortar on the flow table shall be remixed for 30 sec. with that remaining in the mixing bowl. Immediately after remixing, the mortar shall be uniformly distributed without compacting over the sheet of dampened filter paper in

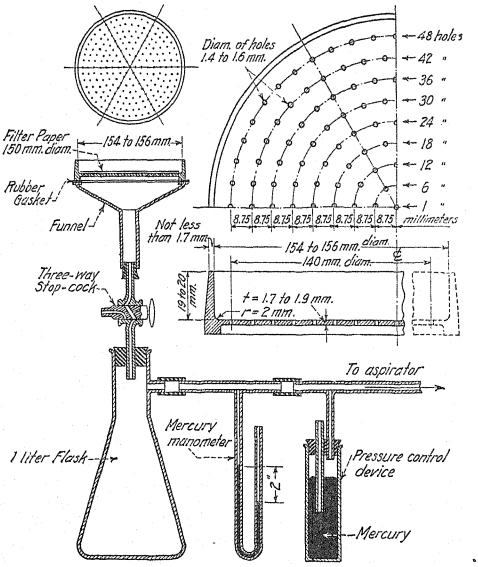


FIG. 1.—Apparatus Assembly for the Water Retention Test.

Schleicher and Schull filter paper No. 575 shall be used. It shall be of such diameter that it will lie flat and completely cover the bottom of the dish.

(*b*) *Method.*—The mortar shall be mixed to a flow of from 100 to 115 per cent in accordance with the procedure described in Section 21. Immediately after making the flow test, the mortar on the flow table shall be remixed for 30 sec. with that remaining in the mixing bowl. Immediately after remixing, the mortar shall be uniformly distributed without compacting over the sheet of dampened filter paper in the perforated dish and the surface leveled off flush with the rim of the dish by drawing a straightedge across the dish with a slightly sawing motion. The dish shall then be seated on the wetted gasket, and with the mercury column set at 2 in., the stopcock shall be turned to apply the vacuum to the

funnel. After suction for 60 sec., the stopcock shall be quickly turned to expose the funnel to atmospheric pressure. The contents of the dish shall then be immediately removed by means of a putty knife or square-end spatula and placed in the mold on the flow table. As each portion of mortar is placed in the mold it shall be well puddled with glove-covered fingers. When the mold is filled, the mortar shall be smoothed off level with the top of the mold, using the edge of a trowel, and the flow determined as previously. The entire operation shall be carried out without interruption and as quickly as possible and shall not require more than 7 min. for completion, starting from the completion of the mixing of the mortar for the first flow determination. Both flow determinations shall be made in accordance with the procedure described in Section 21 (*b*), special care being taken to fill the mold uniformly when obtaining the flow after suction.

Staining Test (Determination of Water-Soluble Alkali)

27. Water-soluble alkali shall be determined in accordance with Section 20 of the Standard Methods of Chemical Analysis of Portland Cement (A.S.T.M. Designation: C 114).[5]

Standard Methods of

CHEMICAL ANALYSIS OF PORTLAND CEMENT[1]

A.S.T.M. Designation: C 114 - 44

ADOPTED 1939; REVISED 1940, 1942, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 114; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods outline procedures for the chemical analysis of portland cement. The procedures appear in the following order:

	Section
Silicon Dioxide	8
Aluminum Oxide and Ferric Oxide	9
Ferric Oxide	10 and 11
Aluminum Oxide	12
Calcium Oxide	13
Magnesium Oxide	14 and 15
Sulfur Trioxide	16
Loss on Ignition	17
Sodium Oxide and Potassium Oxide	18 and 19
Water-Soluble Alkali	20
Phosphorus Pentoxide	21 and 22
Manganic Oxide	23 and 24
Insoluble Residue	25
Chloroform-Soluble Organic Substances	26 and 27
Free Calcium Oxide	28 and 29
Silicon Dioxide, Alternate Method	30
Calcium Oxide, Alternate Method	31 and 32
Magnesium Oxide:	
Alternate Method A	33
Alternate Method B	34 and 35
Alternate Method C	36 and 37
Phosphorus Pentoxide, Alternate Method	38 and 39
Manganic Oxide, Alternate Method	40 and 41
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Sampling

2. Samples of the cement shall be taken and prepared as prescribed in the Standard Methods of Sampling Hydraulic Cement (A.S.T.M. Designation: C 183).[3]

Apparatus

3. (*a*) *Balances.*[4]—The analytical balances used in the chemical determinations shall conform to the following requirements:

Capacity not less than 100 g. in each pan,

The two arms of the beam equal within (*1*) two parts in 100,000 for balances in use or (*2*) one part in 100,000 for new balances (Note 1),

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee C-1 on Cement.

[2] These methods of chemical analysis were editorially rearranged and issued under the designation C 114 in 1939. They formerly constituted a portion of the Standard Methods of Sampling and Testing Portland Cement (A.S.T.M. Designation: C 77) and were published as standard in Methods C 77 from 1930 to 1939.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book

[4] Information on Analytical Balances and Weights, to supplement the requirements for balances and weights prescribed in these methods, has been prepared and is published as information, not a part of these methods; see the compilation of "A.S.T.M. Standards on Cement," issued as a separate publication.

A "tentative revision" of this standard appears in the Tentative Revisions of Standards section at the back of this book

Capable of reproducing results within 0.0002 g., and

Sensibility reciprocal[5] not more than 0.0005 g. per division of the graduated scale for any load up to 100 g. (Note 2). In case of direct-reading balances this requirement does not apply.

Any rapid weighing device that may be provided, such as a chain, damped motion, or heavy riders, shall not introduce errors greater than 0.0001 g. at any reading and with any allowable load on the scale pans.

NOTE 1.—The lengths and ratios of the arms may change with use, and with age even if not used.

NOTE 2.—If an auxiliary metal or glass pan is used, its weight is a part of the load.

(*b*) *Weights.*[4]—The weights used in the chemical determinations shall conform to the requirements of the National Bureau of Standards specifications for class "S" weights as prescribed in Bureau of Standards *Circular No. 3*, "Design and Test of Standards of Mass." The weights (including the riders) shall be checked when questioned, or at least once a year, with weights that have been certified by the National Bureau of Standards within five years. They shall be adjusted within the tolerances given in the above mentioned specifications for class "S" weights, according to their apparent mass as determined by comparison with brass standards in air.

Purity of Water and Reagents

4. (*a*) *Water.*—References to water shall be understood to mean distilled water.

(*b*) *Reagents.*—Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[6] Where such specifications are not available, the best grade available should be used. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

Concentration of Reagents

5. (*a*) *Concentrated Acids and Ammonium Hydroxide.*—When acids and ammonium hydroxide are specified by name or chemical formula only, it shall be understood that concentrated reagents of the following specific gravities or concentrations are intended:

Hydrochloric acid, HCl	sp. gr. 1.18
Hydrofluoric acid, HF	48 per cent
Nitric acid, HNO_3	sp. gr. 1.42
Sulfuric acid, H_2SO_4	sp. gr. 1.84
Ammonium hydroxide, NH_4OH	sp. gr. 0.90

The desired specific gravities or concentrations of all other concentrated acids shall be stated whenever they are specified.

(*b*) *Diluted Acids and Ammonium Hydroxide.*—Concentrations of diluted acids and ammonium hydroxide, except when standardized, shall be specified as a ratio stating the number of volumes of the concentrated reagent to be diluted with a given number of volumes of water, as in the following example: HCl (1:99) means 1 volume of concentrated HCl (sp. gr. 1.18) diluted with 99 volumes of water.

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[5] The sensibility reciprocal is a measure of the sensitiveness of a balance, and is the weight required to move the position of equilibrium of the pointer one division. For a complete definition of sensibility reciprocal, see "Specifications, Tolerances, and Regulations for Commercial Weighing and Measuring Devices," *Handbook H29*, Nat. Bureau Standards, September, 1942, pp. 87 and 88.

[6] "A.C.S. Analytical Reagents," American Chemical Society, Washington, D. C., March, 1941. For later additions and revisions, see "Recommended Specifications for Analytical Reagent Chemicals" by the Committee on Analytical Reagents of the American Chemical Society, *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 16, No. 5, May, 1944, p. 281. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards" by Joseph Rosin, D. Van Nostrand Co., Inc., New York, N. Y., (1937).

(*c*) *Standard Solutions.*—Concentrations of standard solutions shall be expressed as normalities or as equivalents in grams per milliliter of the component to be determined, for example: 0.1 *N* $Na_2S_2O_3$, or $K_2Cr_2O_7$ (1 ml. = 0.004 g. Fe_2O_3).

(*d*) *Nonstandardized Solutions.*—Concentrations of nonstandardized solutions prepared by dissolving a given weight of the solid reagent in a solvent shall be specified in grams of the reagent per liter of solution, and it shall be understood that water is the solvent unless otherwise specified, for example: NaOH (10 g. per l.) means 10 g. of NaOH dissolved in water and diluted with water to 1 liter. In the case of certain reagents the concentration may be specified as a percentage by weight, for example: ethanol (50 per cent) means a solution containing 50 g. of ethanol per 100 g. of solution. Other nonstandardized solutions may be specified by name only, and the concentration of such solutions will be governed by the instructions for their preparation.

Glassware[7]

6. (*a*) All glass apparatus and vessels used in analytical work should be carefully selected to meet the particular requirements for each operation. Standard volumetric flasks, burettes, and pipettes should be of precision grade.

(*b*) New and improved types of glass apparatus are available, including colored glass for the protection of solutions affected by light, alkali-resistant glass for use where superior resistance to alkalies is important, and a high-silica glass having exceptional resistance to thermal shock. Standard-taper, interchangeable, ground-glass joints are useful in analytical work.

Number of Chemical Determinations

7. (*a*) The number of chemical tests shall be as prescribed in Section 6 of the Standard Methods of Sampling Hydraulic Cement (A.S.T.M. Designation: C 183).[3]

(*b*) The chemical determinations shall be made in accordance with the procedures described in the following Sections 8 to 43.

NOTE.—These methods of chemical analysis are not all considered as possessing the highest obtainable accuracy, but are methods to be followed in making acceptance tests on cements covered by specifications requiring that tests be made in accordance with the analytical procedures contained in this standard.

SILICON DIOXIDE

Procedure

8. (*a*) Transfer 0.5 g. of the sample to an evaporating dish, preferably of platinum for the sake of celerity in evaporation, moisten with 10 ml. of cold water to prevent lumping, add 5 to 10 ml. of HCl, and digest with the aid of gentle heat and agitation until solution is complete. Solution may be aided by light pressure with the flattened end of a glass rod. Evaporate the solution to dryness on a steam bath. Without heating the residue any further, treat it with 5 to 10 ml. of HCl and then an equal amount of water, or pour at once upon the residue 10 to 20 ml. of HCl (1:1). Then cover the dish and digest for 10 min. on the bath or a hot plate. Dilute the solution with an equal volume of hot water, immediately filter, and wash the separated

[7] For further information the following references may be consulted:
E. F. Mueller and R. M. Wilhelm, "Methods of Testing Thermometers," *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part 1, p. 493 (1938);
J. J. Moran, "Methods of Testing Volumetric Glassware," *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 492 (1941);
J. P. Bader and E. L. Peffer, "Methods of Testing Hydrometers," *Proceedings*, Am. Soc. Testing Mats., Vol. 42, p. 463 (1942);
E. L. Peffer and G. C. Mulligan, "Testing of Glass Volumetric Apparatus," Nat. Bureau Standards *Circular C 434*, Superintendent of Documents, Government Printing Office, Washington, D. C. (1941).

SiO_2 thoroughly with hot water (Note), and reserve the residue.

NOTE.—The washing of the SiO_2 precipitates can be made more effective by using hot HCl (1:99) and then completing the washing with hot water.

(*b*) Again evaporate the filtrate to dryness, baking the residue in an oven for 1 hr. at 105 to 110 C. Then take up the residue with 10 to 15 ml. of HCl (1:1) and heat on the bath or hot plate. Dilute the solution with an equal volume of hot water and catch and wash the small amount of SiO_2 it contains on another filter paper. Reserve the filtrate and washings for the determination of $Al_2O_3 + Fe_2O_3$ (Section 9).

(*c*) Transfer the papers containing the residues (Paragraphs (*a*) and (*b*)) to a weighed platinum crucible. Dry and ignite the papers, first at a low heat until the carbon of the filter paper is completely consumed without inflaming, and finally at 1100 to 1200 C. until the weight remains constant.

(*d*) Treat the SiO_2 thus obtained, which will contain small amounts of impurities, in the crucible with a few drops of water, about 10 ml. of HF, and one drop of H_2SO_4, and evaporate cautiously to dryness. Finally, heat the small residue at 1050 to 1100 C. for a minute or two, cool, and weigh. The difference between this weight and the weight previously obtained represents the amount of SiO_2. Consider the weighed residue remaining after the volatilization of the SiO_2 as combined aluminum and ferric oxides and add it to the result obtained in the determination of these oxides (Section 9).

(*e*) *Blank.*—Make a blank determination, following the same procedure and using the same amounts of reagents, and correct the results obtained in the analysis accordingly.

(*f*) *Calculation.*—Calculate the percentage of SiO_2 to the nearest 0.1 by multiplying the weight in grams of SiO_2 by 200, (100 divided by the weight of sample used (0.5 g.)).

ALUMINUM OXIDE AND FERRIC OXIDE

Procedure

9. (*a*) To the filtrate reserved in accordance with Section 8 (*b*) (Note 1), which should have a volume of about 250 ml., add HCl if necessary to insure a total of 10 to 15 ml. of the acid. Add a few drops of methyl red indicator (2 g. per liter of ethanol), and heat to boiling. Then treat with NH_4OH (Note 2), drop by drop, until one drop changes the color of the solution to a distinct yellow. Bring to boiling the solution containing the precipitated aluminum and ferric hydroxides, boil for 1 or 2 min., and then filter. Wash the precipitate once by decantation and wash slightly on the filter with hot NH_4Cl (20 g. per l.).

NOTE 1.—If a platinum evaporating dish has been used for the dehydration of SiO_2, iron may have been partially reduced. At this stage, add about 3 ml. of saturated bromine water to the filtrate and boil the filtrate to eliminate the excess bromine before adding the methyl red indicator.

NOTE 2.—The NH_4OH used to precipitate the hydroxides must be free of contamination with CO_2.

(*b*) Set aside the filtrate and transfer the precipitate and filter paper to the same beaker in which the first precipitation was effected. Dissolve the aluminum and ferric hydroxides in hot HCl (1:3), dilute the solution to about 100 ml., and reprecipitate the hydroxides as described in Paragraph (*a*). Filter the solution, and wash the precipitate with two 10-ml. portions of hot NH_4Cl (20 g. per l.). Combine the filtrate and washings with the filtrate set aside and reserve for the determination of CaO (Section 13).

(*c*) Place the precipitate in a weighed platinum crucible, heat slowly until the papers are charred, and finally ignite to constant weight at 1050 to 1100 C. with care to prevent reduction, and weigh as $Al_2O_3 + Fe_2O_3$.

(*d*) Add to the weight of $Al_2O_3 + Fe_2O_3$ any impurities that may be found in the residue of SiO_2 according to Section 8 (*d*). If a determination of phosphorus pentoxide is required and made according to Section 22, the weight of $Al_2O_3 + Fe_2O_3$ shall be corrected for the amount of P_2O_5 found.

(*e*) *Blank.*—Make a blank determination, following the same procedure and using the same amounts of reagents, and correct the results obtained in the analysis accordingly.

(*f*) *Calculation.*—Calculate the percentage of $Al_2O_3 + Fe_2O_3$ to the nearest 0.1 by multiplying the weight in grams of $Al_2O_3 + Fe_2O_3$ by 200, (100 divided by the weight of sample used (0.5 g.)).

FERRIC OXIDE

Reagents

10. (*a*) *Stannous Chloride Solution.*—Dissolve 10 g. of $SnCl_2 \cdot 2H_2O$ in 25 ml. of HCl and 75 ml. of water. Add scraps of iron-free granulated tin and boil until the solution is clear. Keep the solution in a closed dropping bottle containing metallic tin.

(*b*) *Phosphoric-Sulfuric Acid Mixture.*—Add slowly, while stirring, 150 ml. of H_3PO_4 (sp. gr. 1.71) and 150 ml. of H_2SO_4 to 500 ml. of water, and then dilute to 1 liter and mix.

(*c*) *Diphenylamine Indicator.*—Dissolve 1 g. of diphenylamine in 100 ml. of H_2SO_4.

(*d*) *Standard Potassium Dichromate Solution* (*1 ml. = 0.004 g. Fe_2O_3*).—Dissolve 2.457 g. of $K_2Cr_2O_7$ in water and dilute to 1 liter. Standardize against standard iron wire of known iron content in the same manner as directed for the ferric oxide determination in cement (Section 11), using a weight of iron wire that will yield a titration approximately equal to that required by the cement sample in question. Calculate the Fe_2O_3 equivalent of the solution in grams per milliliter by multiplying the weight in grams of iron in the amount of iron wire used by 1.430 (molecular ratio of Fe_2O_3 to 2 Fe) and dividing by the volume in milliliters of $K_2Cr_2O_7$ solution required.

Procedure

11. (*a*) To 1 g. of the sample add 40 ml. of cold water, stir, and add 15 ml. of HCl. Heat the solution, and grind the cement with the flattened end of a glass rod until it is evident that the cement is completely decomposed. Heat the solution to boiling and treat it with the $SnCl_2$ solution, added drop by drop while stirring, until the solution is decolorized. Add one drop in excess and cool the solution to room temperature. Rinse the inside of the vessel with water, and add all at once 10 ml. of a cool, saturated $HgCl_2$ solution. Stir the solution vigorously for 1 min., add 15 ml. of H_3PO_4 - H_2SO_4 mixture and 3 drops of diphenylamine indicator, and dilute to 150 ml. with cold water. Titrate with the standard $K_2Cr_2O_7$ solution. The end point is taken as the point at which a drop causes an intense deep blue coloration that remains unchanged on further addition of the standard $K_2Cr_2O_7$ solution.

(*b*) *Calculation.*—Calculate the percentage of Fe_2O_3 to the nearest 0.1 as follows:

$$Fe_2O_3, \text{ per cent} = EV \times 100$$

where:

E = Fe_2O_3 equivalent of the $K_2Cr_2O_7$ solution in grams per milliliter, and

V = milliliters of $K_2Cr_2O_7$ solution required by the 1-g. sample used.

ALUMINUM OXIDE

Procedure

12. *Calculation.*—Calculate the percentage of Al_2O_3 to the nearest 0.1 by deducting the percentage of Fe_2O_3 determined according to Section 11 from the percentage of $Al_2O_3 + Fe_2O_3$ determined according to Section 9.

NOTE.—Any titanium and phosphorus that may be present will be precipitated with aluminum and ferric hydroxides in the procedure specified in Section 9 and are counted as Al_2O_3. Aluminum oxide should not be corrected for TiO_2 or P_2O_5 unless such a correction is expressly specified or the determination of the component is required. If the precipitations of the hydroxides are made as directed, little manganese, if any, will be retained in the precipitate.

CALCIUM OXIDE

Procedure

13. (*a*) Acidify the combined filtrates obtained in the precipitations of aluminum and ferric hydroxides (Section 9) with HCl and evaporate them to a volume of about 100 ml. Add 40 ml. of saturated bromine water to the hot solution and immediately add NH_4OH until the solution is distinctly alkaline. Boil the solution for 5 min. or more, making certain that the solution is at all times distinctly alkaline. Allow the precipitate to settle, filter, and wash with hot water. Wash the beaker and filter once with HNO_3 (1:33) that has been previously boiled to expel nitrous acid, and finally with hot water. Discard the precipitate. Acidify the filtrate with HCl and boil until all the bromine is expelled. Make the solution alkaline with NH_4OH and bring the solution to boiling. Add to the boiling solution 25 ml. of boiling ammonium oxalate (50 g. per l.), the boiling being continued until the precipitated calcium oxalate assumes a well-defined granular form. Allow the solution to stand for about 20 min. or until the precipitate has settled, filter, and wash moderately with ammonium oxalate (1 g. per l.). Reserve the filtrate and washings.

(*b*) Place the filter paper containing the precipitate, wet, in a platinum crucible and burn off the paper at a low heat. Then ignite the precipitate until the calcium oxalate is converted to CaO, redissolve (Note 1) in HCl, and dilute the solution to 100 ml. with water. Add NH_4OH in slight excess and bring the solution to boiling. If a small amount of hydroxides separates at this point, filter it out, weigh, and add the amount to that found in the original determination of $Al_2O_3 + Fe_2O_3$. Reprecipitate the CaO with ammonium oxalate as before (Note 2), allow to stand until settled, filter, and wash, taking care to avoid an excess of the washing solution. Combine the filtrate with that already obtained and reserve for the determination of MgO (Section 15).

NOTE 1.—Add 5 ml. of cold water cautiously to the first ignited precipitate and then cautiously add 10 ml. of HCl (1:1). As soft-burnt calcium oxide may be too active in slaking, a temperature of 1100 to 1200 C. is recommended for the ignition of the first precipitate. After a little practice it is possible to dissolve hard-burnt calcium oxide in this way without explosive spattering.

NOTE 2.—If the second precipitate of calcium oxalate is so fine as to cause difficulty in filtering, or if the precipitate causes bumping, the volume of solution may be increased to 150 or 200 ml., instead of 100 ml., prior to precipitation.

(*c*) Weigh the precipitate as CaO, after ignition and heating at 1100 to 1200 C. to constant weight in a weighed, covered platinum crucible.

(*d*) *Blank.*—Make a blank determination, following the same procedure and using the same amounts of reagents, and correct the results obtained in the analysis accordingly.

(*e*) *Calculation.*—Calculate the percentage of CaO to the nearest 0.1 by multiplying the weight in grams of CaO by 200, (100 divided by the weight of sample used (0.5 g.)).

MAGNESIUM OXIDE

Reagents

14. *Ammonium Nitrate Wash Solution.*—Dissolve 100 g. of NH_4NO_3 in water, add 200 ml. of NH_4OH, and dilute to 1 liter.

Procedure

15. (*a*) Acidify the filtrates reserved in the determination of CaO (Section 13) with HCl and concentrate to about 150 ml. Add to this solution about 10 ml. of $(NH_4)_2HPO_4$ (250 g. per l.) and cool the solution by placing in a beaker of ice water. After cooling, add NH_4OH drop by drop, while stirring constantly, until the crystalline magnesium ammonium phosphate begins to form, and then in moderate excess (5 to 10 per cent of the volume of the solution), the stirring being continued for several minutes. Set the solution aside for at least 8 hr. in a cool atmosphere, and then filter.

(*b*) Redissolve the precipitate thus obtained in hot HCl (1:4), dilute the solution to about 100 ml., add 1 ml. of $(NH_4)_2HPO_4$ (250 g. per l.), and then add NH_4OH drop by drop, while stirring constantly, until the precipitate is again formed as described and the NH_4OH is in moderate excess. Cool, allow to stand for about 2 hr., filter, and wash with two 10-ml. portions of the NH_4NO_3 wash solution. Place in a weighed platinum crucible, slowly char the paper, and carefully burn off the resulting carbon. Ignite the precipitate at 1100 to 1200 C. to constant weight, taking care to avoid bringing the pyrophosphate to melting.

(*c*) *Blank.*—Make a blank determination, following the same procedure and using the same amounts of reagents, and correct the results obtained in the analysis accordingly.

(*d*) *Calculation.*—Calculate the percentage of MgO to the nearest 0.1 as follows:

$$\text{MgO, per cent} = W \times 72.4$$

where:

W = grams of $Mg_2P_2O_7$, and
72.4 = molecular ratio of 2 MgO to $Mg_2P_2O_7$ (0.362) divided by weight of sample used (0.5 g.) and multiplied by 100.

SULFUR TRIOXIDE

Procedure

16. (*a*) To 1 g. of the sample add 25 ml. of cold water and 5 ml. of HCl. Heat the solution and grind the material with the flattened end of a glass rod until it is evident that decomposition of the cement is complete. Dilute the solution to 50 ml. and digest on a steam bath for 15 min. Filter, and wash the residue thoroughly with hot water. Dilute the solution to 250 ml. and heat to boiling. Add slowly, drop by drop from a pipette, 10 ml. of hot $BaCl_2$ (100 g. per l.) and continue the boiling until the precipitate is well formed. Digest the solution on the steam bath for at least 3 hr., preferably overnight. Filter the precipitate, wash, place the paper and contents in a weighed platinum crucible, and slowly char and consume the paper without inflaming. Then ignite and weigh the $BaSO_4$.

(*b*) *Blank.*—Make a blank determination, following the same procedure and using the same amounts of reagents, and correct the results obtained in the analysis accordingly.

(*c*) *Calculation.*—Calculate the percentage of SO_3 to the nearest 0.1 as follows:

$$\text{SO}_3\text{, per cent} = W \times 34.3$$

where:

W = grams of $BaSO_4$, and
34.3 = molecular ratio of SO_3 to $BaSO_4$ (0.343) multiplied by 100.

(*d*) *Optional Procedure.*—The acid filtrate obtained in the determination of

the insoluble residue (Section 25) may be used for the determination of SO_3 instead of using a separate sample.

LOSS ON IGNITION

Procedure

17. (*a*) Heat 1 g. of the sample in a weighed, covered platinum crucible of 20 to 25-ml. capacity, as follows, using either method A or B as specified.

(*b*) *Method A.*—Place the crucible in a hole in an asbestos board clamped horizontally, so that about three fifths of the crucible projects below the board, and heat at a full red heat (900 to 1000 C.) for 15 min.; check the loss in weight by a second heating for 5 min. Take care to wipe off particles of asbestos that may adhere to the crucible when withdrawn from the hole in the board. Greater neatness and shortening of the time of heating may be secured by making a hole to fit the crucible in a circular disk of sheet platinum and placing this disk over a somewhat larger hole in an asbestos board.

(*c*) *Method B.*—Place the crucible in a muffle furnace at any temperature between 900 and 1000 C. for 15 min., and check the loss in weight by a second heating for 5 min.

(*d*) *Calculation.*—Calculate the percentage of loss on ignition to the nearest 0.1 by multiplying the loss in weight in grams by 100.

SODIUM OXIDE AND POTASSIUM OXIDE

Reagents

18. (*a*) *Chloroplatinic Acid Solution (50 g. per l.).*—Dissolve 5 g. of $H_2PtCl_6 \cdot 6H_2O$ in water and dilute to 100 ml.

(*b*) *Gladding Wash Solution.*—Dissolve 100 g. of NH_4Cl in water, dilute to 500 ml., and add to the solution an amount of K_2PtCl_6 that is more than sufficient to make a saturated solution (about 1 g.). Shake the mixture frequently for 6 to 8 hr., allow it to stand at least 12 hr., and filter. The residue may be used for the preparation of a new supply of the wash solution.

Procedure

19. (*a*) Weigh 5 g. of the sample of finely pulverized cement and mix it with 1 g. of NH_4Cl by grinding the two together in an agate mortar. Add 4 g. of $CaCO_3$ to the mixture and continue the grinding until the ingredients are thoroughly mixed. Place about 0.5 cm. of $CaCO_3$ on the bottom of a 40 to 60-ml. platinum crucible, add the above mixture to the crucible, and cover the mixture with about 0.5 cm. of $CaCO_3$. Mount the crucible through a hole in an asbestos board about 0.3 cm. in thickness, so that the crucible projects about 1 cm. above the board. Cover the crucible with a well-fitting cover and set a platinum crucible containing water on it. The purpose of the second crucible is to prevent the loss of alkali by volatilization, and this crucible must be kept filled with water through the entire period of ignition. Heat the crucible at a very low temperature at first and gradually raise the temperature within 1 hr. to a bright red heat. At no time should the temperature be raised so fast that white NH_4Cl fumes can be seen issuing from the crucible. Finally, ignite the crucible at 900 to 1000 C. for 1 hr.

(*b*) With the aid of hot water, remove the sintered material from the crucible and let the sinter digest with about 150 ml. of water in a casserole on a water bath for about 4 hr. If there are still lumps in the casserole, break them up with a pestle or glass rod. Allow the mixture to digest on the water bath 4 hr. more, or longer. Boil the mixture for 1 min., allow the residue to settle, and decant the clear solution through a

filter paper into a large beaker. Add 30 ml. of water to the casserole, bring the mixture to a boil, allow the residue to settle, and decant the clear filtrate through the same filter paper (Note). Perform three more times this addition of water and decantation. Finally, wash the residue into the filter paper, rinse the casserole, and wash the residue once with hot water.

NOTE.—If it is desired to speed up the work without allowing the residue to settle so as to give a clear solution to decant easily, filter the solution at once, leaving as much of the residue in the dish as possible, discharge the filtrate held in the stem of the funnel by tapping the funnel or lifting the filter paper a little out of the funnel, and wash the residue on the filter paper back into the dish.

(*c*) Combine the filtrates and washings and evaporate them to about 50 ml. Add 3 or 4 g. of $(NH_4)_2CO_3$ to the solution, and boil it for 1 min. Filter the solution, and wash the residue six times with cool $(NH_4)_2CO_3$ (10 g. per l.) that contains a little NH_4OH. Add 10 ml. of ammonium oxalate (50 g. per l.) and 2 or 3 ml. of NH_4OH to the filtrate. Evaporate the solution to about 20 ml., filter into a dish, and wash the residue with hot water. Add 1 ml. of H_2SO_4 to the filtrate and evaporate it to dryness.

(*d*) Ignite the residue in the dish carefully until the excess H_2SO_4 is driven off and then raise the temperature to a dull red heat. Cool the dish, dissolve the residue in 50 ml. of water, and add 2 ml. of ammonium oxalate (50 g. per l.) and 2 or 3 ml. of NH_4OH to the solution. Evaporate the solution to about 10 ml., filter the solution into a platinum dish, and wash the residue with hot water. Add 0.5 ml. of H_2SO_4 to the filtrate and evaporate it to dryness. Ignite the residue, gradually raising the temperature to 600 to 700 C., until all the excess H_2SO_4 and ammonium salts are expelled. Cool the dish, dissolve the residue in a small amount of water, evaporate the solution to dryness, ignite the residue at 600 to 700 C. for 15 min., cool it in a desiccator, and weigh. Dissolve the residue in about 20 ml. of hot water and filter the solution. Wash the filter paper with hot water and put it in the dish. Ignite carefully until the paper is consumed and then continue the ignition at 600 to 700 C. for 10 min. Cool the dish in a desiccator, and weigh. The difference between these two weights represents the weight of the alkali as Na_2SO_4 and K_2SO_4.

(*e*) Add 1 to 2 ml. of HCl to the filtrate in a vessel made of glass, silica, or porcelain and evaporate it to about 10 ml. To the solution, add 6 ml. of the solution of chloroplatinic acid for each 0.10 g. of the mixed sulfates and add 3 ml. in excess. Evaporate the solution on a water bath to a thick sirup, carrying this evaporation as far as possible without allowing the solution to go to dryness. The sirupy solution should solidify on cooling. Add 100 ml. of ethanol (85 per cent) to the solution, while stirring thoroughly, and allow the precipitate of potassium chloroplatinate (K_2PtCl_6) to settle for just 3 hr. Filter the clear liquid through a dried and tared Gooch crucible with a mat of asbestos or a fused-in fritted-glass disk. Wash the precipitate five times by decantation with ethanol (85 per cent) and then rinse the crucible three times with the ethanol. Wash the precipitate three times by decantation with the Gladding wash solution, stirring the precipitate thoroughly each time. Transfer the precipitate to the crucible with the Gladding wash solution, taking care that none remains in the original vessel, and finally wash the crucible and contents six times with ethanol (85 per cent). Dry the crucible at 105 to 110 C. for at least 3 hr. Cool, and weigh it. The difference between

these two weights represents the weight of K_2O as K_2PtCl_6.

(*f*) *Blank.*—Make a blank determination, following the same procedure and using the same amounts of reagents, and correct the results obtained in the analysis accordingly.

(*g*) *Calculation.*—Calculate the percentages of Na_2O and K_2O to the nearest 0.01 as follows:

$$Na_2O, \text{ per cent} = (W_1 - W_2 \times 0.3584) \times 8.728, \text{ and}$$

$$K_2O, \text{ per cent} = W_2 \times 3.874$$

where:

W_1 = grams of $Na_2SO_4 + K_2SO_4$,
W_2 = grams of K_2PtCl_6,
0.3584 = molecular ratio of K_2SO_4 to K_2PtCl_6,
8.728 = molecular ratio of Na_2O to Na_2SO_4 (0.4364) divided by the weight of sample used (5 g.) and multiplied by 100, and
3.874 = molecular ratio of K_2O to K_2PtCl_6 (0.1937) divided by the weight of sample used (5 g.) and multiplied by 100.

WATER-SOLUBLE ALKALI[8]

NOTE.—The determination of water-soluble alkali according to Section 20 should not be considered as a substitute for the determination of total alkali according to Section 19. Moreover, it is not to be assumed that in the procedure of Section 20 all water-soluble alkali is obtained that may be in the cement. A uniform method is essential where there is a specified limit on the content of water-soluble alkali or where several lots of cement are compared on the basis of water-soluble alkali.

Procedure

20. (*a*) Weigh 25 to 150 g. of the sample (Note 1) into a large beaker and add 250 ml. of water to the cement, while stirring thoroughly (Note 2). Let the mixture stand for 30 min. at room temperature, stir it again, and filter it through a Büchner funnel. Return the residue to the original beaker, rinse the funnel with 150 ml. of water into the beaker, stir the mixture thoroughly, and let it stand 30 min. at room temperature. Again stir the solution and filter it through the Büchner funnel. Return the residue to the beaker and rinse the funnel with 100 ml. of water into the beaker. Stir the mixture thoroughly, and let it stand 30 min. at room temperature. Stir it and filter it as before, discard the residue without washing, and complete the determination as described in either Paragraphs (*b*) to (*d*) or Paragraph (*e*) (Note 3).

NOTE 1.—The amount of sample taken for analysis depends on the content of water-soluble alkali, varying from 25 g. for 0.5 per cent or more to 150 g. for 0.1 per cent or less.

NOTE 2.—As the amount of water-soluble alkali obtained depends on the manner in which it is leached out of cement, it is important that the procedure be followed closely.

NOTE 3.—In most cases the procedure described in Paragraphs (*b*) to (*d*) is to be preferred because it is simpler and usually gives all the data necessary.

(*b*) Follow the procedure as described in Section 19 (*c*) and (*d*).

(*c*) *Blank.*—Make a blank determination, following the same procedure and using the same amounts of reagents, and correct the results obtained in the analysis accordingly.

(*d*) *Calculation.*—Calculate as Na_2O the percentage of water-soluble alkali to the nearest 0.01 as follows:

$$Na_2O, \text{ per cent} = \frac{W \times 43.64}{S}$$

where:

W = grams of $Na_2SO_4 + K_2SO_4$,
S = grams of sample used, and

[8] The procedure for determining water-soluble alkali as described in Section 20 is identical in substance with Section F-3b of the Federal Specification for Masonry Cement (SS-C-181b, January 12, 1938) in regard to leaching of cement. The differences in the treatment of the combined filtrates should not produce an appreciable difference in the results.

43.64 = molecular ratio of Na_2O to Na_2SO_4 (0.4364) multiplied by 100.

(*e*) If it is required that water-soluble alkali be reported as water-soluble Na_2O and water-soluble K_2O, follow the procedure as described in Section 19, starting with Paragraph (*c*) and making the necessary changes in the formulas of Paragraph (*g*) due to the weight of sample taken for analysis. If desired, a measured portion of the combined filtrates may be taken for the steps following the leaching in order to avoid the use of a large amount of chloroplatinic acid.

PHOSPHORUS PENTOXIDE

Reagents

21. (*a*) *Ammonium Molybdate Solution.*—Dissolve 100 g. of molybdic acid (85 per cent MoO_3) in a mixture of 400 ml. of water and 100 ml. of NH_4OH and cool the solution to room temperature. While stirring constantly, pour this solution very slowly into a cooled mixture of 600 ml. of water and 400 ml. of HNO_3. No precipitate should form that does not redissolve upon stirring. Add 1 drop of $(NH_4)_2HPO_4$ (250 g. per l.) to the solution, shake it thoroughly, and allow it to stand at least 24 hr. before use. Filter portions of the solution as needed.

(*b*) *Magnesia Mixture.*—Dissolve 50 g. of $MgCl_2 \cdot 6H_2O$ and 100 g. of NH_4Cl in 500 ml. of water. Make the solution slightly alkaline with NH_4OH and allow it to stand at least 12 hr. Filter the solution, acidify it with HCl, and dilute to 1 liter.

Procedure

22. (*a*) Weigh 1 to 3 g. of the sample (Note) and 10 g. of NH_4NO_3 into a 150-ml. beaker. Mix the contents, treat with 10 ml. of HNO_3, and stir quickly, using the flattened end of a glass rod to crush lumps of cement, until the cement is completely decomposed and the thick gel of SiO_2 is broken up. Cover the beaker with a watch glass, place it on a water bath or a hot plate at approximately 100 C. for 15 to 20 min., and stir the contents occasionally during the heating. Add 20 ml. of hot water to the beaker and stir the contents. If the cement contains an appreciable amount of manganese, as shown by the presence of a red or brown residue, add a few milliliters of $NaNO_2$ (50 g. per l.) to dissolve this residue. Boil the contents of the beaker until all nitrous fumes are completely expelled. This should not take more than 5 min., and water should be added to replace any lost by evaporation. Filter into a 500-ml. Erlenmeyer flask under suction and with a platinum cone to support the filter paper, and wash the residue of SiO_2 until the volume of filtrate and washings is about 150 ml.

NOTE.—The amounts of sample and reagents used depend on the content of phosphorus in the cement. The minimum requirements are sufficient if the cement contains 0.5 per cent P_2O_5 or more. The maximum amounts are required if the content of P_2O_5 is 0.1 per cent or less.

(*b*) Heat the solution to 85 C. and add 50 to 100 ml. of the ammonium molybdate solution. Stopper the flask and shake it vigorously for 5 min., then set aside for 30 min. Filter the precipitate, rinsing the flask five times and washing the filter paper eight times with cold KNO_3 (10 g. per l.). Put the original flask under the funnel containing the precipitate. Pour 25 ml. of NH_4OH (1:4) over the precipitate and around the upper edges of the filter paper until the precipitate is entirely dissolved. Wash the paper three times with cold water, twice with HCl (1:1), and finally three times with cold water. Acidify the solution with HCl and add to it 1 ml. of citric acid (350 g. per l.) to hold traces of iron, if any, in solution.

(*c*) Cool the solution to room temperature and transfer it to a 400-ml. beaker. The volume should now be about 150 ml. Add 25 ml. of the magnesia mixture to the solution, and then, while stirring constantly, add drop by drop 50 ml. of NH_4OH at such a rate that 5 min. are required for the addition. Allow the mixture to stand 4 hr. or longer. Filter the precipitate, and wash it a few times with NH_4OH (1:20). Dissolve the precipitate into the original beaker by pouring 25 ml. of HCl (1:1) on the precipitate. Wash the filter paper thoroughly with HCl (1:20). With the washings and water, dilute the solution to 50 to 100 ml., and add 1 to 2 ml. of the magnesia mixture. While stirring constantly, add NH_4OH slowly to the solution as before. Allow the mixture to stand 2 hr. or longer. Filter the precipitate, scrub and rinse the beaker, and wash the precipitate ten times with NH_4OH (1:20). Ignite the precipitate in a weighed crucible at 1100 to 1200 C. to constant weight.

(*d*) *Blank.*—Make a blank determination, following the same procedure and using the same amounts of reagents, and correct the results obtained in the analysis accordingly.

(*e*) *Calculation.*—Calculate the percentage of P_2O_5 to the nearest 0.01 as follows:

$$P_2O_5, \text{ per cent} = \frac{W \times 63.79}{S}$$

where:

W = grams of $Mg_2P_2O_7$,
S = grams of sample used, and
63.79 = molecular ratio of P_2O_5 to $Mg_2P_2O_7$ (0.6379) multiplied by 100.

MANGANIC OXIDE

Reagent

23. *Standard Sodium Arsenite Solution (1 ml. = 0.0003 g. Mn_2O_3).*—Dissolve in 100 ml. of water 3.0 g. of Na_2CO_3 and then 0.90 g. of As_2O_3, heating the mixture until the solution is as complete as possible. If the solution is not clear or contains a residue, filter the solution. Cool it to room temperature, transfer to a volumetric flask, and dilute to 1 liter.

Dissolve 0.58 g. of $KMnO_4$ in 1 liter of water and standardize it against about 0.03 g. of sodium oxalate oxidimetric standard furnished by the National Bureau of Standards (standard sample No. 40) according to the directions furnished with the sodium oxalate. Put 30.0 ml. of the $KMnO_4$ solution in a 250-ml. Erlenmeyer flask. Add 60 ml. of HNO_3 (1:4) and 10 ml. of $NaNO_2$ (50 g. per l.) to the flask. Boil the solution until the HNO_2 is completely expelled. Cool the solution and add sodium bismuthate ($NaBiO_3$), and finish by titrating with the standard $NaAsO_2$ solution in the manner described below for the determination of manganese in cement. Calculate the Mn_2O_3 equivalent of the $NaAsO_2$ solution in grams per milliliter as follows:

$$E = \frac{A \times 7.08}{BC}$$

where:

E = Mn_2O_3 equivalent of the $NaAsO_2$ solution in grams per milliliter,
A = grams of $Na_2C_2O_4$,
B = milliliters of $KMnO_4$ solution required by the $Na_2C_2O_4$ used,
C = milliliters of $NaAsO_2$ solution required by 30.0 ml. of $KMnO_4$ solution, and
7.08 = molecular ratio of Mn_2O_3 to 5 $Na_2C_2O_4$ (0.236) multiplied by 30.0 (milliliters of $KMnO_4$ solution).

Procedure

24. (*a*) Weigh 1.0 to 3.0 g. of the sample (Note 1) into a 250-ml. beaker

and treat it with 5 to 10 ml. of water and then with 60 to 75 ml. of HNO_3 (1:4). Boil the mixture until the solution is as complete as possible. Add 10 ml. of $NaNO_2$ (50 g. per l.) to the solution and boil it until the HNO_2 is completely expelled (Note 2), taking care not to allow the volume of the solution to become so small as to cause the precipitation of gelatinous SiO_2. There may be some separated SiO_2, which may be ignored, but if there is still a red or brown residue, use more $NaNO_2$ (50 g. per l.) to effect a complete decomposition and then boil again to expel the HNO_2. Filter the solution into a 250-ml. Erlenmeyer flask and wash the filter paper with water.

NOTE 1.—The amount of cement taken for analysis depends on the content of manganese, varying from 1 g. for about 1 per cent of Mn_2O_3 to 3 g. for 0.25 per cent or less of Mn_2O_3.

NOTE 2.—When $NaNO_2$ is added, the expulsion of HNO_2 by boiling must be complete. If any HNO_2 remains in the solution, it will react with the added $NaBiO_3$ and decrease its oxidizing value. If there is any manganese in the cement, the first small quantity of $NaBiO_3$ should bring out a purple color.

(*b*) The solution should have a volume of 100 to 125 ml. Cool it to room temperature. To the solution add a total of 0.5 g. of $NaBiO_3$ in small quantities, while shaking intermittently. After the addition is completed, shake the solution occasionally for 5 min. and then add to it 50 ml. of cool HNO_3 (1:33) which has been previously boiled to expel nitrous acid. Filter the solution through a pad of ignited asbestos in a Gooch crucible or a carbon or fritted-glass filter with the aid of suction. Wash the residue four times with the cool HNO_3 (1:33). Titrate the filtrate immediately with the standard solution of As_2O_3. The end point is reached when a yellow color is obtained free of brown or purple tints and it does not change upon further addition of As_2O_3.

(*c*) *Blank.*—Make a blank determination, following the same procedure and using the same amounts of reagents, and correct the results obtained in the analysis accordingly.

(*d*) *Calculation.*—Calculate the percentage of Mn_2O_3 to the nearest 0.01 as follows:

$$Mn_2O_3, \text{ per cent} = \frac{EV}{S} \times 100$$

where:

E = Mn_2O_3 equivalent of the $NaAsO_2$ solution in grams per milliliter,
V = milliliters of $NaAsO_2$ solution required by the sample, and
S = grams of sample used.

INSOLUBLE RESIDUE[9]

Procedure

25. (*a*) To 1 g. of the sample, add 10 ml. of cold water and 5 ml. of HCl. Warm the solution gently and grind the material with the flattened end of a glass rod for a few minutes until it is evident that decomposition of the cement is complete (Note). Dilute the solution to 50 ml. and digest for 15 min. at a temperature just short of boiling. Filter the residue, wash six times with hot water, and digest the filter paper and contents in 100 ml. of NaOH (10 g. per l.), the solution being held at a temperature just short of boiling for 15 min. Using methyl red as indicator, acidify the solution with HCl and add an excess equivalent to 4 or 5 drops of HCl. Filter and wash the residue 12 to 15 times with hot NH_4Cl (20 g. per l.).

[9] This method or any other method designed for the estimation of acid-insoluble substance in any type of cement is empirical, because the amount obtained depends on the reagents and the time and temperature of digestion. If the amount is large, there may be a little variation in duplicate determinations. The directions should be followed closely in order to reduce the variation to a minimum. When the method is used on blended cement, the decomposition in acid is considered to be complete when the portland cement clinker is decomposed completely. An NH_4Cl solution is used in the final washing to prevent finely ground insoluble material from passing through the filter paper.

Ignite the residue in a tared crucible at 900 to 1000 C., cool it in a desiccator, and weigh.

NOTE.—If a sample of portland cement contains an appreciable amount of manganic oxide, there may be brown compounds of manganese which dissolve slowly in cold diluted HCl but rapidly in hot HCl in the specified strength. In all cases, dilute the solution as soon as decomposition is complete.

(*b*) *Blank.*—Make a blank determination, following the same procedure and using the same amounts of reagents, and correct the results obtained in the analysis accordingly.

(*c*) *Calculation.*—Calculate the percentage of insoluble residue to the nearest 0.01 by multiplying the gain in weight in grams by 100.

CHLOROFORM-SOLUBLE ORGANIC SUBSTANCES[10]

Reagent

26. *Chloroform.*—If the blank determination as described in Section 27 (*e*) exceeds 0.0015 g., the chloroform should be distilled before use. Chloroform recovered in the procedure may be slightly acid but can be reused for the portions to be shaken with the aqueous acid solution of the sample in the 1-liter funnel. Chloroform used for washing the filter and transferring the extract should be fresh or distilled from fresh chloroform.

Procedure

27. (*a*) Place 40 g. of cement in a large beaker and mix it with 520 ml. of water, starting with a small portion of the water and stirring the slurry until it is free of lumps and is of a uniform, thick consistency before adding the balance of the water. While stirring the slurry to a uniform suspension, add rapidly 185 ml. of HCl in which 10 g. of $SnCl_2 \cdot 2H_2O$ have been dissolved, and continue the stirring. When the cement is completely decomposed, cool the solution rapidly to room temperature by immersion in cold water, while stirring occasionally. Transfer the solution to a 1-liter Squibb separatory funnel (Note 1) and rinse the beaker with a little water. Add 75 ml. of chloroform to the solution, rinsing the beaker with the chloroform in three portions as it is added. Stopper the funnel, shake it vigorously for 5 min., and allow the water and chloroform to stand 15 min. to separate.

NOTE 1.—The use of grease to lubricate the stopcocks and glass stoppers of the separatory funnels should be avoided. Wetting the stopcocks with water before using will assist in their easy operation.

(*b*) Draw off the lower chloroform layer into a 125-ml. Squibb separatory funnel, including the scum (Note 2) with a few milliliters of the aqueous solution, making certain that all the scum is transferred. Keep the amount of the aqueous layer transferred to an absolute minimum, since excessive water in the 125-ml. funnel may result in incomplete extraction of the scum and may cause an emulsion which does not separate readily. Shake the funnel vigorously to insure the complete extraction of the scum. Allow the chloroform to separate, and draw it into a 250-ml. Squibb separatory funnel which contains 50 ml. of water and a few drops of HCl, making sure to keep the scum behind in the 125-ml. funnel. Shake the 250-ml. funnel, and draw the chloroform into another 250-ml. funnel that contains 50 ml. of water and a few drops of HCl. Shake this funnel as in the case of the

[10] This method was specially designed for the determination of Vinsol resin and tallow in portland cement, although mineral oil, common rosin, calcium stearate, and other fatty acid compounds, and probably some other substances, if present, will be included in the determination. With all the apparatus set up, an experienced operator may be able to complete a determination in about 3 hr. Extreme care is necessary in the entire procedure. The method may be applied to other types of cement besides portland cement, although if the cement contains a large amount of acid-insoluble matter, the emulsions may separate slowly, and less vigorous shaking, more chloroform, and more washing may be necessary.

first 250-ml. funnel. When the chloroform separates, draw it into an Erlenmeyer flask (Note 3), taking care not to allow any water to enter the flask.

NOTE 2.—There is usually a dark colored scum at the liquid interface. It may contain chloroform-soluble organic substance after shaking in the funnel, where the proportion of water to chloroform is great. It may be concentrated and confined to a small volume by gently twirling the funnel after the scum has been drawn into the narrower part of the funnel.

NOTE 3.—The liquid is later distilled. No cork or rubber stoppers should be used. A 250- or 300-ml. soil analysis flask, fitted with a condenser tube by means of a ground joint, is satisfactory. The tube may be bent near the neck and the remaining part fitted with a water-cooling jacket. Chloroform thus recovered may be reused as directed in Section 26.

(*c*) Rinse the original large beaker with 25 ml. of chloroform, and add the liquid to the 1-liter separatory funnel. Carry out the operations as described above, retaining the original wash water in the 250-ml. funnels. Repeat, using another 25-ml. portion of chloroform.

(*d*) Distill the combined chloroform extracts in the Erlenmeyer flask until their volume is reduced to 10 to 15 ml. Filter the remaining liquid into a weighed 100-ml. glass beaker or platinum dish (Note 4) through a small filter paper that has been washed with chloroform. Rinse the flask and wash the paper with several small portions of chloroform. Evaporate the extract at a low temperature (not over 63 C.) to dryness (Note 5) and heat it in an oven at 57 to 63 C. for 3 min. Pass dry air into the vessel for 15 sec., cool, and weigh. Repeat the heating and weighing until two successive weighings do not differ by more than 0.0010 g. The higher of the last two weights is taken as the true weight.

NOTE 4.—A platinum dish is preferable, as it quickly attains the temperature of the balance. If a glass beaker is used, it should be allowed to stand in the case of the balance for at least 20 min. before the weighing.

NOTE 5.—Care should be taken in drying the extract, as many of the chloroform-soluble organic substances are somewhat volatile when heated for a long time at even moderate temperatures. With protection from the accumulation of dust, the solution may be evaporated at room temperature overnight.

When a quick evaporation is desired, the solution may be evaporated on a hot plate at a low heat under a stream of dry air through a glass tube (about 1 cm. in inside diameter) until it is about 0.3 cm. in depth. Then remove the vessel from the hot plate and continue a slow stream of dry air until the residue appears dry. Then continue with a more rapid stream of dry air for 5 min. at room temperature before placing the vessel in the oven at 57 to 63 C. After each 3-min. heating period in the oven, pass dry air into the vessel for about 15 sec. before weighing. The air may be dried by passing it through a cheap desiccant, such as calcium chloride or sulfuric acid, followed by a desiccant of high efficiency, such as magnesium perchlorate or anhydrous calcium sulfate, with care taken to avoid the carrying of dust from the desiccant by the air. Instead of using compressed air, which is often contaminated with oil, dirt, and moisture, one can place the chloroform solution under a bell glass and induce a stream of air through the desiccants by means of an aspirator or vacuum pump.

(*e*) *Blank.*—Make a blank determination, using 40 g. of the cement which is ignited at 950 to 1000 C. for 1 hr. (Note 6) and reground, following the same procedure and using the same amounts of reagents, and correct the results obtained in the analysis accordingly.

NOTE 6.—Care should be taken to completely burn off the organic substance. A 100-ml. flat platinum dish, in which the sample is well spread out, and a muffle furnace are advised for this purpose. If such a furnace is not available, a large high-temperature burner of the Meker type may be used. Thorough stirring of the sample should be done frequently—every 5 min. when a burner is used.

(*f*) *Calculation.*—Calculate the percentage of chloroform-soluble organic substances to the nearest 0.001 by mul-

tiplying the weight in grams of residue (Note 7) by 2.5, (100 divided by the weight of sample used (40 g.).

NOTE 7.—If the organic substance in the cement is tallow, the residue is the fatty acids resulting from the hydrolysis of the tallow in the hot acid solution, and its weight should be multiplied by 1.05 to give the weight of the original glycerides in the tallow. If the original substance is calcium stearate, the residue is stearic acid, and its weight multiplied by 1.07 gives the weight of calcium stearate.

FREE CALCIUM OXIDE IN PORTLAND CEMENT AND CLINKER

Reagents

28. (*a*) *Phenolphthalein Indicator.*—Dissolve 1 g. of phenolphthalein in 100 ml. of absolute ethanol, and neutralize the solution with NaOH dissolved in absolute ethanol.

(*b*) *Glycerol-Ethanol Solvent.*—Prepare a solution consisting of 1 part by volume of glycerol (Note 1) and 5 parts by volume of absolute ethanol (Note 2). To each liter of this solution add 2 ml. of the indicator. The solvent should be slightly alkaline (Note 3) to the indicator. If the solvent is colorless, add a dilute solution of NaOH in absolute ethanol until a slight pink color appears. If the initial color is pink, remove it with the standard ammonium acetate solution (Paragraph (*c*)) and add the NaOH solution until a slight pink color appears. Heat 60 ml. of the solvent to boiling. If the pink color persists, add one small drop of the standard solution of ammonium acetate (0.02 ml., equivalent to 0.0001 g. CaO) to the hot solvent. If the pink color does not disappear, the solvent contains too much alkali, which should be reduced. The solvent, if allowed to stand for a considerable length of time, may become slightly acid and should be checked and, if necessary, readjusted from time to time. (Note 4.)

NOTE 1.—The glycerol should conform to the requirements for glycerol of the Committee on Analytical Reagents of the American Chemical Society.[6] Water is not usually included by the manufacturer in the list of impurities on the label but is usually present. The water content should not exceed about 5 per cent. The test should be made with a pycnometer at 25 C. and the specific gravity at 25/25 C. should be not less than 1.249.

NOTE 2.—Absolute ethanol may be replaced by anhydrous ethanol denatured according to Formula No. 3a or 2b of the U. S. Bureau of Internal Revenue. The alcohol is 95 per cent ethanol and 5 per cent methanol according to Formula No. 3a or 99.5 per cent ethanol and 0.5 per cent benzol according to Formula No. 2b.

NOTE 3.—While a solvent that is exactly neutral at the boiling point is the ideal, it is difficult to prepare due to the end point not being sharp and the danger of adding too much ammonium acetate. The error due to slight alkalinity is not more than 0.01 per cent in a determination of free CaO and may be disregarded. In preparing the solvent, the end point may be better observed if the solvent is put in a tall cylinder placed on white paper. The solvent may be observed from the top and the color matched with that of acidified solvent in a similar cylinder.

NOTE 4.—Glycerol and ethanol are highly hygroscopic. Every effort should be made to avoid exposing them and other materials unnecessarily to water and CO_2 in air. Bottles with an outlet near the bottom or fitted with a siphon are convenient. They can be filled to the top and protected with tubes containing soda lime. Condensers may also be fitted with such tubes at the top.

(*c*) *Standard Ammonium Acetate Solution (1 ml. = 0.005 g. CaO).*—Prepare a standard solution of ammonium acetate in absolute ethanol as follows: Dissolve 16 g. of crystalline ammonium acetate (Note 5) in 1 liter of ethanol. Standardize this solution by titrating against pure CaO that is *freshly* prepared by calcining pure calcium carbonate or calcium oxalate in a platinum crucible at 900 to 1000 C. to constant weight. When the calcined CaO has cooled in a desiccator, perform the following operations in quick succession: grind it in an agate mortar, weigh out 0.05 to 0.06 g. into a dry 200- or 250-ml. Erlenmeyer

flask, and add 60 ml. of the glycerol-ethanol solvent to the flask (Note 6). Disperse the CaO in the solution by shaking the flask and attach a reflux condenser (Note 7). Boil the mixture. The ebullition should be positive but not so violent as to cause bumping or excessive evaporation. Then remove the condenser and immediately titrate the solution, while near boiling, with the standard ammonium acetate solution. Replace the condenser and boil the solution again. Repeat the titrations at intervals (Note 8). Shake the flask frequently between titrations to shorten the time required for the boiling. The titration is complete when no further color appears in the solution during continuous boiling for 1 hr. (Note 9). Calculate the CaO equivalent of the ammonium acetate solution in grams per milliliter by dividing the weight of CaO used by the volume of solution required.

NOTE 5.—Ammonium acetate is generally sold in a damp condition and should be as dry as possible. It may be dried over a drying agent for two weeks or more.

NOTE 6.—Sometimes pure CaO will cake on the bottom of the flask, particularly if the glycerol is anhydrous. This trouble may be avoided by putting a few grams of clean, dry quartz sand in the flask before the introduction of CaO and solvent.

NOTE 7.—If a new stopper is used and has white powder on it, it should be thoroughly washed before using. A glass tube about 6 mm. in diameter and 50 to 100 cm. in length may be used as an air-cooled condenser, but a short condenser is not recommended because a loss of alcohol is liable to occur through evaporation. In such a case it is necessary to add neutralized ethanol from time to time to maintain the same proportion of glycerol to ethanol. If the proportion is greater than one to three, a partial decomposition of cement may take place. Instead of an ordinary flask, plain tubing, and a stopper, a flask with a ground-in glass stopper and condenser tube may be used, preferably one with a standard-taper joint. A water-cooled condenser is the most satisfactory and it may be as short as 1 ft. Some loss of alcohol may also occur through dripping from the condenser during the titrations and should be compensated for by the addition of neutralized ethanol.

NOTE 8.—In general the intervals may be 20 min. but they will chiefly depend on the rapidity of the dissolution of CaO. They may be 5 to 10 min. in the beginning and then be increased to 30 min. or 1 hr. in the end. If the solution contains a large amount of dissolved CaO and its boiling is continued a long time without titration, crystals (probably calcium glyceride) may form which dissolve slowly and increase the time required for the completion of the titration.

NOTE 9.—The end point will not be the same for a hot solution and a cold solution, especially when a large amount of calcium acetate is present. The titration should always be carried out while the solution is boiling hot. If the end point is determined accurately, the solution will turn pink upon cooling, and this can serve as evidence that the end point has not been greatly overstepped.

Procedure

29. (*a*) Weigh 1 g. of finely ground clinker or cement (Note 1) into a flask, add 60 ml. of the solvent to the flask, and proceed as in the standardization of the ammonium acetate solution (Section 28 (*c*)). The end point is considered to be reached when the content of free CaO in the sample does not increase by more than 0.05 per cent during the last 2 hr. of boiling (Note 2).

NOTE 1.—The method is designed especially for fresh clinker. As the method does not differentiate between free CaO and free $Ca(OH)_2$, any free $Ca(OH)_2$ that may be present will be included in the determination and calculated as free CaO. The method may be applied to cement or aged clinker, if desired, but the possibility of the presence of free $Ca(OH)_2$ should be kept in mind.

The sample should be fine enough to pass completely through a standard No. 200 (74-micron) sieve. If it is not, take about 1.2 g. of the sample and sieve it with a small clean No. 200 (74-micron) sieve. Grind the residue in an agate mortar until it is fine enough to pass through the sieve. Mix all the screenings and ground residue thoroughly, grind slightly over 1 g. rapidly in the agate mortar for 2 or 3 min., and take 1 g. for the determination of free CaO. Avoid unnecessary exposure of the sample

to the atmosphere. If the sample is fine enough to pass completely through the sieve, grind slightly over 1 g. as above before weighing 1 g. for the determination.

NOTE 2.—No excess of ammonium acetate should be added at any time because an excess of it may react with calcium aluminate and silicates. To avoid such excess, use a small burette or measuring pipette fitted with a glass stopcock or a rubber pinchcock and small tip which delivers about 50 drops per milliliter. If the titrations are far apart and the reagent evaporates in the tip, a few drops may be discarded before each titration and the drops used in titration may be counted and converted in terms of milliliters. It is a good precaution to allow a slight pink color to remain in the solution after each of the early titrations.

Vigorous boiling is more essential with cement than with pure CaO and, if possible, should be so conducted that it is not necessary to shake the flask vigorously at intervals. The flask should not be shaken vigorously while it is disconnected from the condenser because the solvent, if superheated, may boil, expel alcohol vapor, and then draw in air carrying water and CO_2. A good procedure is to bring the solution and sample to boiling rapidly over a bunsen flame in the beginning before placing the flask on a hot plate and connecting it with the condenser. If there is a tendency to bump, it may be reduced by adding glass beads or pure quartz sand to the flask or putting a thin mat of asbestos between the flask and the hot plate.

If in the final titrations there is doubt as to the exact end point, add a drop of the indicator to the quiescent liquid in the flask and carefully observe the point where the indicator strikes the liquid. If no color appears, the end point has been reached. If the sample settles during boiling and leaves a partially clear layer of solution, another way to overcome difficulty in discerning a change in the color of the solution is to swirl the flask after the addition of the solution of ammonium acetate so as to avoid the dispersion of the sample. A strong daylight lamp with a reflector may be used as an aid in the discernment of the end point. The contents of the flask may be matched with similar contents in another flask which contains an excess of ammonium acetate.

If it is necessary to leave the determination uncompleted, remove the flask, add just enough ammonium acetate to discharge the pink color, and stopper the flask tightly. When renewing the determination, boil the mixture before repeating the titrations.

(*b*) *Calculation.*—Calculate the percentage of free CaO to the nearest 0.1 as follows:

$$\text{Free CaO, per cent} = EV \times 100$$

where:

E = CaO equivalent of the ammonium acetate solution in grams per milliliter, and

V = milliliters of ammonium acetate solution required by the sample.

ALTERNATE METHODS

NOTE.—The alternate methods described in Sections 30 to 43, inclusive, are provided for those who wish to use procedures shorter or more convenient than the preceding ones for the routine determination of certain components. In case of dispute, results obtained according to Sections 8 to 29, inclusive, shall govern.

SILICON DIOXIDE

Alternate Method

Procedure

30. (*a*) Mix thoroughly 0.5 g. of the sample and about 0.5 g. of NH_4Cl in a 50-ml. beaker, cover the beaker with a watch glass, and add cautiously 5 ml. of HCl, allowing the acid to run down the lip of the covered beaker. After the chemical action has subsided, lift the cover, add one or two drops of HNO_3, stir the mixture with a glass rod, replace the cover, and set the beaker on a water bath for 30 min. (Note 1). During this time of digestion, stir the contents occasionally and break up any remaining lumps to facilitate the complete decomposition of the cement. Fit a medium-texture filter paper to a funnel, transfer the precipitate to the filter as completely as possible without dilution, and allow the solution to drain through. Scrub the beaker with a rubber policeman and rinse the beaker and policeman.

Wash the filter two or three times with hot HCl (1:99) and then with ten or twelve small portions of hot water, allowing each portion to drain through completely. Reserve the filtrate and washings for the determination of $Al_2O_3 + Fe_2O_3$ (Note 2).

NOTE 1.—A hot plate may be used instead of a water bath if the heat is so regulated as to approximate that of a water bath.

NOTE 2.—Determine $Al_2O_3 + Fe_2O_3$ by the procedure described in Section 9. Reserve the combined filtrates for the determination of CaO (Section 13 or 32).

(*b*) Transfer the filter paper and residue to a weighed platinum crucible, dry, and ignite, at first slowly until the carbon of the paper is completely consumed without inflaming and finally at 1050 to 1100 C. for 1 hr. Weigh the residue as SiO_2.

(*c*) *Blank.*—Make a blank determination, following the same procedure and using the same amounts of reagents, and correct the results obtained in the analysis accordingly.

(*d*) *Calculation.*—Calculate the percentage of SiO_2 to the nearest 0.1 by multiplying the weight in grams of SiO_2 by 200, (100 divided by the weight of the sample used (0.5 g.)).

CALCIUM OXIDE

Alternate Method

NOTE.—See Note immediately preceding Section 30.

Reagents

31. *Standard Potassium Permanganate Solution (0.18 N).*—Prepare a solution of $KMnO_4$ on the basis of 5.64 g. per l. The solution should not be filtered through any filter containing organic matter. It is most convenient to siphon off clear solution without disturbing the sediment on the bottom of the bottle. Standardize the solution against 0.7500 g. of sodium oxalate oxidimetric standard furnished by the National Bureau of Standards (standard sample No. 40) according to the directions furnished with the sodium oxalate. If the burette reading is 62.77 ml., the solution contains 0.0056357 g. of $KMnO_4$ per milliliter, which is equivalent to exactly 0.005 g. of CaO. Because of impurities and deterioration, the reading in the standardization is usually not 62.77 ml. Calculate the CaO equivalent of the solution in grams per mill liter as follows:

$$E = \frac{0.31385}{V}$$

where:

E = CaO equivalent of the $KMnO_4$ solution in grams per milliliter,

V = milliliters of $KMnO_4$ solution required by 0.7500 g. $Na_2C_2O_4$, and

0.31385 = 62.77 multiplied by 0.005.

NOTE.—The solution is intended to be equivalent to 0.005 g. of CaO per milliliter, or 1 per cent CaO in a 0.5-g. sample for each milliliter. The numbers 0.0056357 and 62.77 are obtained as follows: 0.005 g. of CaO multiplied by the molecular ratio of 2 $KMnO_4$ to 5 CaO gives 0.0056357 g. of $KMnO_4$. 0.75 g. of $Na_2C_2O_4$ multiplied by the molecular ratio of 2 $KMnO_4$ to 5 $Na_2C_2O_4$ gives 0.35375 g. of $KMnO_4$. 0.35375 g. divided by 0.0056357 g. per ml. gives 62.77 ml.

Procedure

32. (*a*) Add a few drops of NH_4OH to the combined filtrates obtained in the determination of $Al_2O_3 + Fe_2O_3$, (Note 2, Section 30), and bring the solution to boiling. Add to the boiling solution 25 ml. of boiling ammonium oxalate (50 g. per l.) and continue the boiling for 5 to 10 min.; then allow the solution to stand for 15 to 20 min. Filter, and wash the precipitate eight to ten times with hot water, the total amount of water used in rinsing the beaker and washing

not to exceed 75 ml. Reserve the filtrate for the determination of MgO (Section 33). Wash the precipitate back into the original beaker, and add 200 ml. of water and 10 ml. of H_2SO_4 (1:1). Heat the solution just short of boiling, and titrate it with the standard $KMnO_4$ solution (Note 1) to a permanent pink color. Add the filter paper and macerate it. Continue the titration slowly until the pink color is permanent for 10 sec.

NOTE 1.—The temperature of the standard $KMnO_4$ solution should not vary from its standardization temperature so much as to cause a serious error in the determination of CaO. At ordinary room temperatures the volume of pure water changes to the extent of 0.01 to 0.04 per cent for each degree Centigrade, depending on the temperature.

(*b*) *Blank.*—Make a blank determination, following the same procedure and using the same amounts of reagents (Note 2), and correct the results obtained in the analysis accordingly.

NOTE 2.—When the amount of calcium oxalate is very small, its oxidation by $KMnO_4$ is slow to start. Before the titration, add a little $MnSO_4$ to the solution to catalyze the reaction.

(*c*) *Calculation.*—Calculate the percentage of CaO to the nearest 0.1 as follows:

$$\text{CaO, per cent} = EV \times 200$$

where:

E = CaO equivalent of the $KMnO_4$ solution in grams per milliliter,
V = milliliters of $KMnO_4$ solution required by the sample, and
200 = 100 divided by the weight of sample used (0.5 g.).

MAGNESIUM OXIDE

NOTE.—Alternate method A may be used when it is desired to determine MgO in the filtrate from the determination of CaO (Section 32). Alternate method B or C may be used when the determinations of SiO_2, Al_2O_3, Fe_2O_3, and CaO are omitted.

Alternate Method A

Rapid Gravimetric Determination

NOTE.—See Note immediately preceding Section 30.

Procedure

33. (*a*) Acidify the filtrate from the determination of CaO (Section 32) with HCl and evaporate by boiling to about 250 ml. Cool the solution to room temperature, and add 10 ml. of $(NH_4)_2HPO_4$ (100 g. per l.) and 30 ml. of NH_4OH. Stir the solution vigorously during the addition of NH_4OH and then for 10 to 15 min. longer. Let the solution stand for at least 8 hr. in a cool atmosphere and filter. Wash the residue five or six times with NH_4OH (1:20) and ignite in a tared crucible, at first slowly until the filter paper is charred and then burned off, and finally at 1050 to 1100 C. for 30 to 45 min. Weigh the residue as $Mg_2P_2O_7$.

(*b*) *Blank.*—Make a blank determination, following the same procedure and using the same amounts of reagents, and correct the results obtained in the analysis accordingly.

(*c*) *Calculation.*—Calculate the percentage of MgO to the nearest 0.1 as follows:

$$\text{MgO, per cent} = W \times 72.4$$

where:

W = grams of $Mg_2P_2O_7$, and
72.4 = molecular ratio of 2 MgO to $Mg_2P_2O_7$ (0.362) divided by the weight of sample used (0.5 g.) and multiplied by 100.

Alternate Method B

Rapid Determination by Alkalimetry

NOTE.—See Note immediately preceding Section 30.

Reagents

34. (*a*) *Trinitrobenzene Indicator.*—Dissolve 1.0 g. of 1-3-5-trinitrobenzene in 100 ml. of ethanol (95 to 100 per cent).

(*b*) *Phenolphthalein Indicator.*—Dissolve 0.4 g. of phenolphthalein in 100 ml. of ethanol (50 per cent).

(*c*) *Standard Sodium Hydroxide Solution (0.33 N).*—Dissolve 13.0 g. of NaOH in water, add 10 ml. of a freshly filtered, saturated $Ba(OH)_2$ solution, and dilute to 1 liter with water that has been recently boiled and cooled. Shake the solution from time to time for several hours, and filter into a tubulature bottle fitted with an automatic burette. Protect the solution from CO_2 in the air, with soda-lime tubes at the top of the bottle and the burette.

Standardize against acid potassium phthalate or benzoic acid acidimetric standards furnished by the National Bureau of Standards (standard samples Nos. 84 and 39), using the methods in the certificates accompanying the standard samples. Determine the exact normality of the solution, and calculate its MgO equivalent on the basis that 1 ml. of 1 *N* NaOH is equivalent to 0.02016 g. of MgO.

(*d*) *Standard Acid Solution (0.33 N).*—Dilute 32.0 ml. of HCl or 26.0 ml. of HNO_3 to 1 liter with distilled water. Standardize against the standard NaOH solution, using phenolphthalein indicator, and adjust by dilution until the two standard solutions of alkali and acid are of equal strength.

(*e*) *Potassium Iodide-Starch Paper.*—Mix equal volumes of KI (50 g. per l.) and soluble starch solution (10 g. per l.), and dip strips of filter paper into the mixture.

Procedure

35. (*a*) To 5.0 g. of the sample of cement in a 600-ml. beaker, add about 50 ml. of water and 50 ml. of HCl (1:1) and boil until the cement is completely in solution, excepting possibly some SiO_2 which may separate out (Note 1). Add 5 ml. of saturated bromine water and continue the boiling until the excess bromine is completely expelled, as indicated by the absence of a blue coloration on potassium iodide-starch paper when held in the issuing steam. Dilute the solution to about 250 ml. with hot water and add NaOH (200 g. per l.) to the boiling solution from a pipette until the excess acid is neutralized and a permanent precipitate just forms. Continue the boiling and carefully add HCl (1:1) from a pipette until the precipitate dissolves and the solution assumes a yellow $FeCl_3$ color, avoiding much excess of acid. Wash down the sides of the beaker with hot water and continue the boiling for 1 or 2 min., and then remove the beaker from the burner.

NOTE 1.—Some samples of cement, particularly those containing considerable iron or manganese, may require longer boiling or the addition of slightly more HCl to effect complete solution.

(*b*) Cautiously add to the solution 4.0 g. of $CaCO_3$ (weighed to the nearest 0.1 g.). This amount should be sufficient to completely neutralize the acid, with some excess. Dilute the solution to about 400 ml. with hot water and boil vigorously for about 10 min. Remove the beaker from the burner or hot plate, and from a burette carefully add $KMnO_4$ (5 g. per l.) to the hot solution, while stirring vigorously, until the supernatant liquid shows a faint pink color after settling a moment. Avoid the use of more $KMnO_4$ than necessary to produce a faint pink color. Continue the boiling for 2 min., add 2.0 ml. of ethanol, and continue the boiling for a few minutes or until the pink color has disappeared (Note 2).

NOTE 2.—The purpose of the addition of $KMnO_4$ is to remove manganese from the sample,

which otherwise would be precipitated and reported as MgO. The $KMnO_4$ in a hot solution oxidizes the manganese to insoluble MnO_2 and is itself reduced to MnO_2. The ethanol precipitates the excess $KMnO_4$ as MnO_2. In samples containing little or no manganese, this portion of the procedure may be omitted.

(*c*) Transfer the contents of the beaker to a 500-ml. volumetric flask, cool to room temperature, dilute to the mark, and mix by shaking. Filter the solution through dry paper and, after discarding the first 10 or 20 ml. of filtrate, transfer 200 ml. of the filtrate with a pipette to a 500-ml. Erlenmeyer flask. Heat the solution in the flask to boiling and add 3.0 ml. of trinitrobenzene indicator. Add standard NaOH solution, 1.0 ml. at a time, with intermittent boiling for a few seconds, until the magnesium is completely precipitated and a definite pink color persists on boiling. Record the total amount of NaOH used (Note 3).

NOTE 3.—Enough standard NaOH solution should be added to completely precipitate the magnesium without too large an excess which might cause some precipitation of calcium. A guide to proper procedure is the amount of back-titration with acid, which should be held between 1.0 and 2.0 ml. (after being doubled). A little experience with the color change of the trinitrobenzene indicator will enable the operator to hold the back-titration between these limits.

(*d*) Transfer the solution to a 250-ml. volumetric flask, cool to room temperature, dilute to the mark, and mix by shaking. Filter the solution through a dry paper into a graduated cylinder, discarding the first 10 or 20 ml. of filtrate, until 125 ml. of the filtrate are secured. Transfer 125 ml. of the filtrate to a flask, add a few drops of phenolphthalein indicator, and titrate the solution with standard acid solution to the disappearance of the pink color.

(*e*) *Blank.*—Make a blank determination, following the same procedure and using the same amounts of reagents, and correct the results obtained in the analysis accordingly.

(*f*) *Calculation.*—If the acid and alkali solutions are of equal strength, the quantity of alkali in milliliters minus twice the quantity of acid in milliliters is equivalent to the MgO in a 2-g. sample. Calculate the percentage of MgO to the nearest 0.1 as follows:

$$\text{MgO, per cent} = E\,(V_1 - 2V_2) \times 50$$

where:

E = MgO equivalent of the standard alkali in grams per milliliter,
V_1 = milliliters of standard NaOH solution used,
V_2 = milliliters of standard acid used, and
50 = 100 divided by the weight of sample (2 g.) involved in the titration.

Alternate Method C

Rapid Determination by Titration of Magnesium Oxyquinolate

NOTE.—See Note immediately preceding Section 30.

Reagents

36. (*a*) *8-Hydroxyquinoline Solution.*—Dissolve 25 g. of 8-hydroxyquinoline in 60 ml. of glacial acetic acid. When the solution is complete, dilute to 2 liters with cold water. One milliliter of this solution is equivalent to 0.0016 g. of MgO.

(*b*) *Standard Sodium Thiosulfate Solution (0.1 N).*—Dissolve 25 g. of $Na_2S_2O_3 \cdot 5H_2O$ in 200 ml. of water and dilute to 1 liter. Standardize this solution either on sodium oxalate through a $KMnO_4$ solution, or on As_2O_3 through a solution of resublimed iodine. Standard samples Nos. 40 and 83 of the National Bureau of Standards are convenient for this purpose. One milliliter of exactly 0.1 *N* $Na_2S_2O_3$ is equivalent to 0.000504 g. of MgO.

(*c*) *Standard Potassium Bromate - Potassium Bromide Solution (0.2 N).*—Dissolve 20 g. of KBr and 5.57 g. of $KBrO_3$ in 200 ml. of water and dilute to 1 liter. Obtain the ratio of the strength of this solution to that of the standard $Na_2S_2O_3$ solution as follows: To 200 ml. of water in a 400-ml. beaker add exactly 25 ml. of the standard $KBrO_3$ – KBr solution. Add 20 ml. of HCl, stir, and add immediately 10 ml. of KI (250 g. per l.). Mix well and titrate at once with the $Na_2S_2O_3$ solution until nearly colorless. Add 2 ml. of starch solution, and titrate to the disappearance of the blue color. Calculate the ratio in strength of the standard $KBrO_3$ – KBr solution to the standard $Na_2S_2O_3$ solution by dividing the volume of $Na_2S_2O_3$ solution by the volume of $KBrO_3$ – KBr solution used in the titration.

(*d*) *Starch Solution.*—To 500 ml. of boiling water add a cold suspension of 5 g. of soluble starch in 25 ml. of water, cool, add a cool solution of 5 g. of NaOH in 50 ml. of water, add 15 g. of KI, and mix thoroughly.

Procedure

37. (*a*) To 0.5 g. of the sample of cement in a 400-ml. beaker add 10 ml. of water and 10 ml. of HCl. Heat gently and grind any coarse particles with the flattened end of a glass rod until decomposition is complete. Add hot water to make a total volume of 100 ml.

(*b*) Add 3 drops of methyl red indicator (2 g. per liter of ethanol) to the solution and then add NH_4OH until the solution is distinctly yellow. Heat this solution to boiling and boil for 1 to 2 min. Remove from the burner or hot plate and allow to stand until the precipitate has settled. Filter the solution without delay and wash the precipitate twice with hot NH_4Cl (20 g. per l.). Transfer the precipitate with the filter paper to the beaker and dissolve in 10 ml. of HCl (1:1). Dilute to about 100 ml. and heat to boiling. Reprecipitate, filter, and wash the hydroxides as above. Add 1 ml. of NH_4OH to the filtrate. Heat the filtrate to boiling and, while boiling, add 25 ml. of hot ammonium oxalate (50 g. per l.) and continue the boiling for 2 min. Continue the digestion on the steam bath for 10 to 15 min.

(*c*) Cool the solution to 70 C. and add 10 to 25 ml. of the 8-hydroxyquinoline reagent (Note 1) and then 4 ml. of NH_4OH per 100 ml. of solution. Stir the solution on a mechanical stirring machine for 15 min. and set aside until the precipitate has settled (Note 2). Filter the solution and wash the precipitate with hot NH_4OH (1:40). Dissolve the precipitate in 50 to 75 ml. of hot HCl (1:9), dilute the resulting solution to 200 ml., and add 15 ml. of HCl. Cool the solution to 25 C. and add 10 to 35 ml. of the standard $KBrO_3$ – KBr solution (Note 3) from a pipette. Stir the solution and allow to stand for about 30 sec. to insure complete bromination. Add 10 ml. of KI (250 g. per l.). Stir the resulting solution well and then titrate with the standard $Na_2S_2O_3$ solution until the color of the iodine becomes faintly yellow. At this point add 2 ml. of the starch solution and titrate the solution to the disappearance of the blue color.

NOTE 1.—An excess of the 8-hydroxyquinoline reagent should be used. If it is small the result of the determination of MgO may be low; on the other hand, if it is large the result may be high. The following guide may be used.

Approximate Content of MgO, per cent	Approximate Amount of Reagent Required, ml.
0 to 1.5	10
1.5 to 3.0	15
3.0 to 4.5	20
4.5 to 6.0	25

NOTE 2.—The precipitate should be filtered within an hour. Prolonged standing may cause high result.

NOTE 3.—The amount of the standard $KBrO_3$ – KBr solution used may be as follows:

Approximate Content of MgO, per cent	Amount of Standard $KBrO_3$ – KBr Solution, ml.
0 to 1	10
1 to 2	15
2 to 3	20
3 to 4	25
4 to 5	30
5 to 6	35

(*d*) *Blank.*—Make a blank determination, following the same procedure and using the same amounts of reagents, and correct the results obtained in the analysis accordingly.

(*e*) *Calculation.*—Calculate the percentage of MgO to the nearest 0.1 as follows (Note 4):

$$\text{MgO, per cent} = E\,(V_1R - V_2) \times 200$$

where:

E = MgO equivalent of the $Na_2S_2O_3$ solution in grams per milliliter,
V_1 = milliliters of $KBrO_3$ – KBr solution used,
R = ratio in strength of the $KBrO_3$ – KBr solution to the $Na_2S_2O_3$ solution,
V_2 = milliliters of $Na_2S_2O_3$ solution used, and
200 = 100 divided by the weight of sample used (0.5 g.).

NOTE 4.—V_1R represents the volume of $Na_2S_2O_3$ solution equivalent to the volume of $KBrO_3$ – KBr solution used. V_2 represents the amount of $Na_2S_2O_3$ required by the excess $KBrO_3$ – KBr which is not reduced by magnesium oxyquinolate.

PHOSPHORUS PENTOXIDE

Alternate Method

NOTE.—See Note immediately preceding Section 30.

Reagents

38. (*a*) *Ammonium Molybdate Solution.*—Prepare the solution in accordance with Section 21 (*a*).

(*b*) *Standard Sodium Hydroxide Solution (0.3 N).*—Dissolve 12 g. of NaOH in 1 liter of water that has been freshly boiled to expel CO_2 and cooled. Add 10 ml. of a freshly filtered, saturated solution of $Ba(OH)_2$. Shake the solution frequently for several hours, and filter it. Protect it from contamination by CO_2 in the air. Standardize the solution against standard acid potassium phthalate (standard sample No. 84) or benzoic acid (standard sample No. 39) furnished by the National Bureau of Standards, according to the directions furnished with the standard. Calculate the P_2O_5 equivalent (Note) of the solution in grams per milliliter as follows:

$$E = N \times 0.003086$$

where:

E = P_2O_5 equivalent of the NaOH solution in grams per milliliter,
N = normality of the NaOH solution, and
0.003086 = P_2O_5 equivalent of 1 N NaOH in grams per milliliter.

NOTE.—The value of the solution is based on the assumption that the phosphorus in cement is precipitated as $2(NH_4)_3PO_4 \cdot 12MoO_3$ and that the precipitate reacts with the NaOH solution thus:

$$2(NH_4)_3PO_4 \cdot 12MoO_3 + 46NaOH = 2(NH_4)_2HPO_4 + (NH_4)_2MoO_4 + 23Na_2MoO_4 + 22H_2O.$$

The number 0.003086 is obtained by dividing the molecular weight of P_2O_5 (141.96) by 46 (for 46 NaOH in the equation) and by 1000 (number of milliliters in 1 liter).

As the actual composition of the precipitate is influenced by the conditions under which the precipitation is made, it is essential that all the details of the procedure are followed closely as prescribed.

(*c*) *Standard Sulfuric Acid Solution (0.15 N).*—Dilute 4.0 ml. of H_2SO_4 to 1 liter with water that has been freshly boiled and cooled. Standardize against the standard NaOH solution. Deter-

mine the ratio in strength of the standard H_2SO_4 solution to the standard NaOH solution by dividing the volume of NaOH solution by the volume of H_2SO_4 solution used in the titration.

Procedure

39. (*a*) Weigh 1 to 3 g. of the sample (Note 1) and 10 g. of NH_4NO_3 into a 150-ml. beaker. Mix the contents, add 10 ml. of HNO_3, and stir quickly, using the flattened end of a glass rod to crush lumps of cement, until the cement is completely decomposed and the thick gel of SiO_2 is broken up. Cover the beaker with a watch glass, place it on a water bath or a hot plate at approximately 100 C. for 15 to 20 min., and stir the contents occasionally during the heating. Add 20 ml. of hot water to the beaker and stir the contents. If the cement contains an appreciable amount of manganese, as shown by the presence of a red or brown residue, add a few milliliters of $NaNO_2$ (50 g. per l.) to dissolve this residue. Boil the contents of the beaker until all nitrous fumes are completely expelled. This should not take more than 5 min., and water should be added to replace any lost by evaporation. Filter into a 400-ml. beaker under suction and with a platinum cone to support the filter paper, and wash the residue of SiO_2 until the volume of filtrate and washings is about 150 ml.

NOTE 1.—The amounts of sample and reagents used depend on the content of phosphorus in the cement. The minimum requirements are sufficient if the cement contains 0.5 per cent P_2O_5 or more. The maximum amounts are required if the content of P_2O_5 is 0.1 per cent or less.

(*b*) Heat the solution to 69 to 71 C., remove it from the heat source, and immediately add 50 to 100 ml. of the ammonium molybdate solution. Stir the solution vigorously for 5 min., wash down the sides of the beaker with cool KNO_3 solution (10 g. per liter of water freshly boiled to expel CO_2 and cooled), cover the beaker with a watch glass, and allow to stand 2 hr. Using suction, filter the precipitate (Note 2), decanting the solution with as little disturbance to the precipitate as possible. Stir the precipitate in the beaker with a stream of the cool KNO_3 solution, decant the liquid, then wash the precipitate onto the filter. Scrub the stirring rod and beaker with a "policeman," and wash the contents onto the filter. Wash the precipitate until it is acid-free (Note 3), allowing each portion of wash solution to be sucked completely through before adding the next.

NOTE 2.—The filter may be a small ordinary filter paper supported by a platinum cone, or a small Hirsch funnel may be used with filter paper cut to fit and a thin mat of paper pulp or acid-washed asbestos pulp. The filtration should be carried out with care to avoid any loss of the precipitate. The filter should fit well, and the suction should be started before the filtration and maintained until the end of the washing.

NOTE 3.—About ten washings are usually required. Test the tenth washing with one drop of neutral phenolphthalein indicator (see Paragraph (*c*)) and half a drop of the standard NaOH solution. If a definite pink color lasts at least 5 min., the precipitate is considered to be acid-free; otherwise, continue the washing.

(*c*) Transfer the filter and precipitate to the beaker in which the precipitation took place, using small damp pieces of paper to wipe out the funnel and to pick up portions of the precipitate that may remain on it. Add 20 ml. of cool CO_2-free water to the beaker, and break up the filter by stirring rapidly with the "policeman" that was used to scrub the beaker. Add an excess of the standard NaOH solution, stir the contents until all trace of yellow has disappeared, wash down the stirring rod and sides of the beaker with 50 ml. of cool, CO_2-free water, and add 2 drops of neutral

phenolphthalein indicator (10 g. per liter of ethanol). Treat the solution with a measured quantity of the standard H_2SO_4 solution, sufficient to destroy completely the pink color. Complete the titration with the NaOH solution until there is a definite faint pink color that lasts at least 5 min.

(*d*) *Blank.*—Make a blank determination, following the same procedure and using the same amounts of reagents, and correct the results obtained in the analysis accordingly.

(*e*) *Calculation.*—Calculate the percentage of P_2O_5 to the nearest 0.01 as follows:

$$P_2O_5, \text{ per cent} = \frac{E(V_1 - V_2 R)}{S} \times 100$$

where:

E = P_2O_5 equivalent of the NaOH solution in grams per milliliter,
V_1 = milliliters of NaOH solution used,
V_2 = milliliters of H_2SO_4 solution used,
R = ratio in strength of the H_2SO_4 solution to the NaOH solution, and
S = grams of sample used.

MANGANIC OXIDE

Alternate Method

NOTE.—See Note immediately preceding Section 30.

Reagent

40. *Standard Potassium Permanganate Solution (0.18 N).*—Prepare a solution of $KMnO_4$ on the basis of 5.64 g. per l. The solution should not be filtered through any filter containing organic matter. It is most convenient to siphon off clear solution without disturbing the sediment on the bottom of the bottle. Standardize the solution against 0.7500 g. of sodium oxalate oxidimetric standard furnished by the National Bureau of Standards (standard sample No. 40) according to the directions furnished with the sodium oxalate. Calculate the Mn_2O_3 equivalent of the solution in grams per milliliter as follows:

$$E = \frac{0.2650}{A}$$

where:

E = Mn_2O_3 equivalent of the $KMnO_4$ solution in grams per milliliter,
A = milliliters of $KMnO_4$ solution required by 0.7500 g. of $Na_2C_2O_4$, and
0.2650 = 0.7500 multiplied by the molecular ratio of 3 Mn_2O_3 to 10 $Na_2C_2O_4$ (0.3534).

NOTE.—The 0.18 *N* solution of $KMnO_4$ specified in Section 31, which is generally used in the routine volumetric determination of CaO in cement and raw materials, is convenient to use. Exactly 0.005 g. of CaO is equivalent to 0.004224 g. of Mn_2O_3.

Procedure

41. (*a*) Place 2 g. of the sample in a 250-ml. beaker and add about 50 ml. of water to the cement. Stir the mixture until it is in suspension and then add about 15 ml. of HCl. Heat the mixture gently until the solution is as complete as possible. Add 5 ml. of HNO_3 and 50 ml. of water to the solution and boil it until most of the chlorine has been expelled. If necessary, add hot water to maintain the solution at a volume of about 100 ml. Stop the boiling and add ZnO powder to the solution until the acid is neutralized. Add an excess of 3 to 5 g. of ZnO powder to the solution and boil it for a few minutes.

(*b*) Without filtering, and while keeping the solution hot (90 to 100 C.) by intermittent or continuous heating, titrate the solution with the standard $KMnO_4$ solution until a drop of it gives

a permanent pink color (Note). When the end point is approached, add the standard solution drop by drop. After each drop, stir the solution, allow the precipitate to settle a little, and observe the color of the upper stratum of the solution by looking through the side of the beaker.

NOTE.—In the case of a cement in which the approximate content of Mn_2O_3 is unknown, a preliminary determination may be made with rapid titration, 0.5 to 1 ml. of the standard solution being added at a time, and without an attempt to keep the solution close to the boiling point.

(*c*) *Blank.*—Make a blank determination, following the same procedure and using the same amounts of reagents, and correct the results obtained in the analysis accordingly.

(*d*) *Calculation.*—Calculate the percentage of Mn_2O_3 to the nearest 0.01 as follows:

$$Mn_2O_3, \text{ per cent} = EV \times 50$$

where:

E = Mn_2O_3 equivalent of the $KMnO_4$ solution in grams per milliliter,
V = milliliters of $KMnO_4$ solution used, and
50 = 100 divided by the weight of sample used (2 g.).

FREE CALCIUM OXIDE

Alternate Method

NOTE.—See Note immediately preceding Section 30.

Reagents

42. (*a*) *Phenolphthalein Indicator.*—Prepare the indicator as described in Section 28 (*a*).

(*b*) *Glycerol-Ethanol Solvent.*—Prepare the solvent as described in Section 28 (*b*). Either anhydrous $BaCl_2$ or NaCl is used to accelerate the solution of free CaO. If anhydrous $BaCl_2$ is used, dissolve it in the solvent on the basis of 1 g. per 60 ml. of solvent before the solvent is neutralized (Note). If NaCl is used, add 0.5 g. of it to the flask that contains solvent and freshly prepared CaO or sample at the beginning of the standardization or determination as the case may be.

NOTE.—A convenient way to prepare a large amount of solvent is to heat $BaCl_2 \cdot 2H_2O$ at 120 to 130 C. for several hours, dissolve it in hot glycerol (100 to 125 C.) without grinding, and mix the solution with ethanol. If the salt is dehydrated at a much higher temperature, it may be slow to dissolve in hot glycerol or solvent, even when ground.

(*c*) *Standard Ammonium Acetate Solution (0.2 N).*—Prepare and standardize a solution of ammonium acetate as described in Section 28 (*c*), using an accelerator as specified in Paragraph (*b*) of this section.

Procedure

43. Determine free CaO in clinker or cement as directed in Section 29, with the following exceptions:

(*1*) Use anhydrous $BaCl_2$ or NaCl as directed in Section 42 (*b*).

(*2*) The end point is considered to be reached when the content of free CaO does not increase by more than 0.05 per cent during the last hour of boiling (Note).

NOTE.—If the boiling is prolonged too long, the sample may be partially decomposed and the end point may be obscured by colored decomposition products. This alternate method has a tendency to give higher results than the method described in Section 29. Results by the alternate method should be occasionally checked with the other method.

REPORT

Recommended Order for Reporting Analysis

44. The following order is recom-

mended for reporting the results of chemical analysis of portland cement:

Major Components:

SiO_2.....Silicon dioxide
Al_2O_3.....Aluminum oxide
Fe_2O_3.....Ferric oxide
CaO.....Calcium oxide
MgO.....Magnesium oxide
SO_3.....Sulfur trioxide
Loss on ignition

Minor Components:

Na_2O.....Sodium oxide
K_2O.....Potassium oxide
P_2O_5.....Phosphorus pentoxide
Mn_2O_3.....Manganic oxide

Separate Determinations:

Insoluble residue
Chloroform-soluble organic substances
Free calcium oxide
Water-soluble alkali

Standard Methods of

SAMPLING HYDRAULIC CEMENT[1]

A.S.T.M. Designation: C 183 – 44

ADOPTED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 183; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods cover the procedures for sampling hydraulic cement.

Size and Number of Samples; By Whom Taken

2. (*a*) Samples for purpose of tests shall weigh at least 4 lb. each when they are to be composited. Individual test samples, on which all specified tests are to be made, shall weigh at least 8 lb.

(*b*) Test samples shall be either individual or composite samples, as may be specified, and one test sample shall represent not more than 2000 bags unless otherwise specified by the purchaser.

(*c*) The sampling shall be done by or under the direction of a responsible representative of the purchaser.

Sampling from Cars, Trucks, etc.

3. When sampling cement in cars, trucks, boats, warehouses, etc., one sample shall be taken from each 50 bags or portion thereof in the lot and combined to form one test sample. In the case of samples from trucks where the cement is being trucked from one mill, it is permissible to combine the samples from several trucks to form a test sample representing not more than 2000 bags. When bulk shipments are sampled, representative samples shall be taken from well-distributed points.

Sampling from Bulk Storage

4. The cement may be sampled at bulk storage points by any of the methods described in the following Paragraphs (*a*) to (*c*):

(*a*) *From the Conveyor Delivering to Storage.*—One sample of 4 lb. or more shall be taken from at least each 2000 bags passing over the conveyor, except that the sample shall not represent more than 6 hr. of cement production. This may be secured by taking the entire test sample at a single operation, known as the "grab method," or by combining several portions taken at regular intervals, known as the "composite method." When obtaining a composite sample, it shall be secured by combining approxi-

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee C-1 on Cement.

[2] These methods formerly constituted a portion of the Standard Methods of Sampling and Physical Testing of Portland Cement (C 77 – 40), discontinued in 1944, which latter standard was originally part of the Standard Specifications for Portland Cement (A.S.T.M. Designation: C 9) published as standard from 1904 to 1930, at which time the specifications and methods were separated and published individually.

mately equal weights of the cement taken at regular intervals, each portion representing approximately 40 bags. Automatic samplers may be used in obtaining samples.

(*b*) *From Storage at Points of Discharge.*—Sufficient cement shall be drawn from the discharge openings to obtain samples representative of the cement, as determined by the appearance at the discharge openings of indicators placed on the surface of the cement directly above these openings before the drawing of the cement is started. One 4-lb. sample shall be taken for at least each 2000 bags, by either the "grab" or "composite" method as described in Paragraph (*a*).

(*c*) *From Storage by Means of Proper Sampling Tubes.*—When the methods described in Paragraphs (*a*) and (*b*) cannot be applied and when the depth of the cement to be sampled does not exceed 10 ft., samples may be obtained by proper tubes inserted vertically to the full depth of the cement. Samples so taken shall be obtained from points well distributed over the area of storage.

Preparation of Sample

5. (*a*) Samples shall be packed by the purchaser in moisture-proof, airtight containers, which shall be crated, if necessary, by the manufacturer and shipped at the expense of the purchaser. Before testing, samples shall be thoroughly mixed and then passed through a No. 20 (840-micron) sieve in order to break up lumps and remove foreign materials.

(*b*) Composite samples for the tests as required in Section 6 shall be prepared by arranging all test samples in groups, each group representing the number of bags required by the test or tests for which the composite sample is intended. From each of the test samples in a group, equal portions shall be taken, sufficient in amount to form a composite sample large enough to permit making the required physical or chemical determinations. The composite sample thus prepared shall be thoroughly mixed before using.

Number of Tests

6. (*a*) All physical tests required by the purchase specifications shall be made on each test sample taken from cars and trucks (Section 3), with no test sample representing more than 2000 bags.

(*b*) Physical tests on samples taken from bins, boats, warehouses, etc. (Section 4) shall be required as follows:

(*1*) *Time of Setting*, each test sample representing each 2000 bags.

(*2*) *Air Content* (A.S.T.M. Specifications C 175),[3] composite sample representing each 4000 bags.

(*3*) *Fineness* (surface area or 200-mesh fineness as required by the purchase specifications), composite sample representing each 4000 bags.

(*4*) *Strength*, composite sample representing each 4000 bags.

(*5*) *Soundness* (autoclave expansion or steam pat test as required by the purchase specifications), composite sample representing each 12,000 bags.

(*c*) Chemical determinations shall be required as follows:

(*1*) *Sulfur Trioxide* (SO_3), composite sample representing each 4000 bags.

(*2*) *All Specified Chemical Determinations (Except* SO_3), composite sample representing each 20,000 bags.

(*d*) When the total number of bags sampled is less than that specified for any one of the above test or composite samples, all of the respective physical and chemical tests shall be made on the quantity sampled.

[3] Tentative Specifications for Air-Entraining Portland Cement for Concrete Pavements (A.S.T.M. Designation: C 175), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Method of Test for

AUTOCLAVE EXPANSION OF PORTLAND CEMENT[1]

A.S.T.M. Designation: C 151 - 43

ADOPTED, 1943.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C151; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test is intended for determining the soundness of portland cement by means of an autoclave test on a 1 by 1-in. neat cement specimen.

Apparatus

2. (*a*) *Scales.*[2a]—The scales and weights used for weighing materials shall conform to the requirements prescribed in Section 2 (*a*) and (*b*) of the Standard Method of Test for Normal Consistency of Hydraulic Cement (A.S.T.M. Designation: C 187).[3]

(*b*) *Glass Graduates.*—Glass graduates of 150-ml. capacity used for measuring the mixing water shall be made to deliver the indicated volume at 20 C. (68 F.). The permissible variation on these graduates shall be plus or minus 1 ml. The main graduation lines of the cylinders shall be circles and shall be numbered. The least graduations shall extend at least one seventh of the way around, and intermediate graduations shall extend at least one fifth of the way around the cylinder.

(*c*) *Molds.*—Molds shall provide for 1 by 1-in. test specimens of 10-in. effective gage length. The effective gage length shall be considered as that length between the innermost points of the metal inserts used as reference points. The parts of the molds shall be tight fitting and firmly held together when assembled. The molds shall be made of steel or hard metal not readily attacked by the cement paste. There shall be sufficient material in the sides of the molds to prevent spreading or warping. Each end plate of the molds shall be equipped to hold properly in place, during the setting period, a stainless steel or noncorroding metal reference point having a diameter of $\frac{1}{4}$ in. The reference points shall be set so that their principal axes coincide with the principal axis of the test specimen, and shall extend into the specimen $\frac{5}{8}$ in. The distance between the inner ends of the reference points shall be 10 ± 0.1 in. The distance between opposite faces of the molds shall be 1 ± 0.03 in. The height of the molds, measured separately for each specimen compartment, shall be 1 ± 0.03

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-1 on Cement.

[2] Prior to adoption as standard, this method was published as tentative from 1940 to 1943, being revised in 1943.

[2a] This paragraph was editorially revised in 1944.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

in., both for new molds and for molds in use.

(*d*) *Autoclave.*—The autoclave shall consist of a high-pressure steam boiler equipped with an automatic pressure control and a safety valve. The capacity of the heating unit shall be such that with maximum load (water plus specimens) the pressure of the saturated steam in the autoclave may be raised to a gage pressure of 295 psi. (absolute pressure about 310 psi.) in 1 to 1½ hr. from the time the heat is turned on. The automatic pressure control shall be capable of maintaining the pressure at 295 ± 10 psi. for at least 3 hr. A gage pressure of 295 ± 10 psi. corresponds to a temperature of 215.7 ± 1.7 C. (420.3 ± 3 F.). The autoclave shall be designed to permit the pressure to drop from 295 psi. to less than 10 psi. in 1 hr. after the heat supply has been shut off. It shall be equipped with a vent valve for allowing the escape of air during the early part of the heating period and for releasing any steam pressure remaining at the end of the 1-hr. cooling period.

(*e*) *Length Comparator.*—Changes in length of the test specimen shall be measured by a dial gage or micrometer comparator having a range of at least 0.3 in. The instrument shall be graduated to at least 0.001 in., and when tested at any point throughout its range, the error shall not be greater than plus or minus 0.002 in. The difference between repeated measurements shall not be greater than 0.001 in. The comparator shall be equipped with a steel reference bar and shall be frequently checked with this reference bar.

Temperature and Humidity

3. (*a*) The temperature of the molding room and dry materials shall be maintained at not less than 20 C. (68 F.) and not more than 27.5 C. (81.5 F.). The temperature of the mixing water and of the moist closet or moist room shall not vary from 21 C. (70 F.) by more than 1.7 C. (3 F.).

(*b*) The moist closet or moist room shall be so constructed as to provide storage facilities for test specimens at a relative humidity of not less than 90 per cent.

Preparation of Test Specimens

4. (*a*) *Preparation of Molds.*—Molds shall be thinly covered with mineral oil; after this operation the stainless steel or noncorroding metal reference points shall be set, care being taken to keep them clean and free of oil.

(*b*) *Mixing Cement Paste.*[2a]—The standard batch shall consist of 500 g. of cement with sufficient water to give a paste of normal consistency, and shall be mixed in accordance with the procedure described in Section 4 (*a*) of the Standard Method of Test for Normal Consistency of Hydraulic Cement (A.S.T.M. Designation: C 187).[3]

(*c*) *Molding Specimens.*—Immediately following completion of mixing, the test specimen shall be molded in one or two layers, each layer being compacted with the thumbs or forefingers by pressing the paste into the corners, around the reference inserts, and along the surfaces of the molds until a homogeneous specimen is obtained. After the top layer has been compacted, the paste shall be cut off flush with the top of the mold and the surface smoothed with a few strokes of the trowel. During the operations of mixing and molding, the hands shall be protected by rubber gloves.

(*d*) *Storage of Test Specimens.*—After the mold has been filled, it shall be immediately placed in the moist closet or moist room. Specimens shall remain in the molds in the moist closet or moist room for at least 20 hr.; if removed from

the molds before 24 hr., they shall be kept in the moist closet or moist room until tested.

Procedure

5. (*a*) At 24 hr. ± 30 min. after molding, the specimens shall be removed from the moist atmosphere, measured for length, and placed in the autoclave at room temperature in a rack so that the four sides of each specimen will be exposed to saturated steam. The autoclave shall contain enough water to maintain an atmosphere of saturated steam vapor during the entire period of test. Ordinarily, 7 to 10 per cent of the volume of the autoclave should be occupied by the water.

(*b*) To permit air to escape from the autoclave during the early portion of the heating period, the vent valve shall be left open until steam begins to escape. The valve shall then be closed and the temperature of the autoclave shall be raised at such a rate as will bring the gage pressure of the steam to 295 psi. in 1 to 1½ hr. from the time the heat is turned on. The 295 ± 10 psi. pressure shall be maintained for 3 hr. At the end of the 3-hr. period the heat supply shall be shut off and the autoclave cooled at a rate such that the pressure will be less than 10 psi. at the end of 1 hr., and any pressure remaining shall be slowly released by partially opening the vent valve until atmospheric pressure is attained. The autoclave shall then be opened and the test specimens immediately placed in water the temperature of which is above 90 C. (194 F.). The water surrounding the bars shall then be cooled at a uniform rate by adding cold water so that the temperature of the water will be lowered to 21 C. (70 F.) in 15 min. The water surrounding the specimens shall then be maintained at 21 C. (70 F.) for an additional 15 min., when the specimens shall be surface dried and their lengths again measured.[4]

Calculation

6. The difference in length of the test specimen before and after autoclaving shall be calculated to the nearest 0.01 per cent of the effective gage length and shall be reported as the autoclave expansion of the cement. A contraction (negative expansion) shall be indicated by prefixing a minus sign to the percentage expansion reported.

Retests

7. Cement failing to meet the test for soundness in the autoclave may be accepted if it passes a retest, using a new sample, at any time within 28 days thereafter. The provisional acceptance of the cement at the mill shall not deprive the purchaser of the right of rejection on a retest of soundness and time of setting at the time of delivery of the cement to the purchaser.

[4] If it is preferred to make all measurements at 26.5 C. (80 F.), it is recommended that upon removal of the specimens from the moist closet or moist room, they be placed in water maintained at 26.5 C. (80 F.) for at least 15 min., removed and measured for length, and then placed in the autoclave. Upon removal from the autoclave, the specimens and water shall be cooled to 26.5 C. (80 F.) in 15 min. and the specimens kept in water at this temperature for an additional 15 min., and then measured for length.

Standard Method of Test for

FINENESS OF PORTLAND CEMENT BY THE TURBIDIMETER[1]

A.S.T.M. Designation: C 115 - 42

ADOPTED, 1942.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 115; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test covers the Wagner turbidimeter[3] apparatus and procedure for determining the fineness of portland cement as represented by specific surface expressed as total surface area in square centimeters per gram of cement.

APPARATUS

Nature of Apparatus

2. The Wagner turbidimeter consists essentially of a source of light of constant intensity adjusted so that approximately parallel rays of light pass through a suspension of the cement to be tested and impinge upon the sensitive plate of a photoelectric cell. The current generated by the cell is measured with a microammeter and the indicated reading is a measure of the turbidity of the suspension. General considerations indicate that turbidity is in turn a measure of the surface area of the suspended sample of cement. The apparatus, illustrated in Fig. 1, shall consist specifically of the parts described in Sections 3 to 9.

Turbidimeter

3. The turbidimeter shall be mounted in a suitable case and shall include the following features:

(*a*) *Source of Light.*—The source of light (Fig. 1) shall consist of a 3 to 6 candlepower, concentrated-filament electric lamp operated by a 6-v. storage battery, or a source of constant emf. A parabolic reflector shall be mounted behind the lamp and the lamp focused in such manner that approximately parallel rays of light will pass through the sedimentation tank and will impinge upon the photoelectric cell. The light intensity shall be regulated by means of two rheostats having resistances of approximately 6 and 30 ohms, respectively, and mounted in series with the lamp but in parallel with each other.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-1 on Cement.

[2] Prior to adoption as standard, this method was published as tentative from 1934 to 1942, being revised in 1938 and 1941.

[3] This turbidimeter was developed by L. A. Wagner, Research Associate of the Cement Reference Laboratory, National Bureau of Standards, Washington, D. C. A description of the apparatus and mathematical derivations of formulas used are given in the paper, "A Rapid Method for the Determination of the Specific Surface of Portland Cement," by L. A. Wagner, *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 553 (1933).

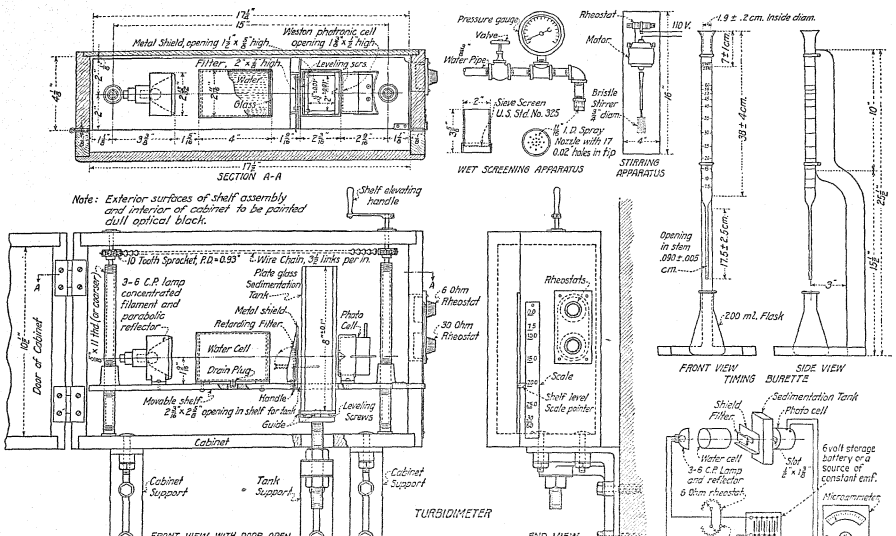


FIG. 1.—Dimensional Details of Turbidimeter Fineness Test Apparatus.

(*b*) *Water Cell.*—The light shall pass through a water cell before entering the sedimentation tank in order that radiant heat from the beam shall be absorbed. The cell shall consist of 3-in. diameter brass tubing, 4 in. in length, with glass windows cemented in the ends with a suitable sealing material. A stoppered hole shall provide for filling with distilled water. The cell shall be so arranged that all rays of light entering the sedimentation tank shall first pass through the water.

(*c*) *Retarding Filter.*—A light-retarding glass or other device shall be provided that will reduce the intensity of light from that corresponding to 100 microamperes to a reading of 20 to 30 microamperes. The light intensity shall be uniformly retarded over the entire area of that portion of the cell which is exposed to light during a test.

(*d*) *Sedimentation Tank.*—The sedimentation tank shall be constructed of $\frac{3}{16}$-in. plate glass cemented together to form a rectangular tank having inside dimensions 2 by $1\frac{1}{2}$ in. by 8 in. in height. The permissible variations on the inside dimensions of the tank shall be plus or minus 0.03 in. in length and width and plus or minus 0.1 in. in height. The 2-in. faces of the tank shall be placed normal to the beam of light, and shall be equidistant within 0.01 in. at all points. A mark shall be placed on the side of the tank to indicate a volumetric content of 335 ml., which is the level to which the tank will be filled in a test.

(*e*) *Photoelectric Cell.*—The means of measuring the light intensity shall be a sensitive photoelectric cell (Weston Photronic type) connected directly to a microammeter. A hood with a horizontal slot $\frac{1}{2}$ in. in height by $1\frac{3}{8}$ in. in width shall be mounted over the face of the photoelectric cell.

(*f*) *Shield.*—A metallic shield having a slot $\frac{5}{8}$-in. in height by $1\frac{1}{2}$ in. in width, as indicated in Fig. 1, shall be placed between the water cell and the sedimentation tank.

(*g*) *Elevating Device.*—The source of light, water cell, photoelectric cell, retarding filter, and shield shall be mounted on a movable shelf which may be raised or lowered, and which may be readily and accurately adjusted so that the turbidity of the suspension may be determined at any desired depth. The sedimentation tank shall be mounted on a base which is independent of the rest of the apparatus so that the tank shall be free from vibration caused by moving the shelf. The level of the light beam with reference to the surface of the suspension shall be indicated by a pointer which will travel along a scale mounted on the cabinet. The zero of the scale shall indicate that position at which the center lines of the slots for the light beam are at the same elevation as the surface of the liquid in the tank when filled to the 335-ml. level. The lines on the scale to be marked 7.5, 10, 15, 20, 25, and 30–60, shall be located at distances from the zero mark equal to values "h" in Table I, and shall indicate the positions at which the pointer should be located when turbidity readings for these particle sizes are taken. The interior of the turbidimeter cabinet and the exterior surfaces of the shelf, parabolic reflector,

TABLE I.—VALUES OF h, d, AND $\frac{h}{d^2}$ TO BE USED IN CALIBRATION OF THE TURBIDIMETER APPARATUS.

Particle Diameter, d, microns	Depth of Suspension, h, cm.	$\frac{h}{d^2}$
60	15	0.00417
55	15	0.00496
50	15	0.00600
45	15	0.00741
40	15	0.00938
35	15	0.01225
30	15	0.01667
25	13.1	0.0210
20	10.0	0.0250
15	6.6	0.0293
10	3.3	0.0333
7.5	2.1	0.0375

water cell, shield, and photoelectric cell hood shall be painted with a dull optical black paint.

Microammeter

4. The microammeter shall have a range of 0 to 50 microamperes, shall be readable to 0.1 microampere, and accurate to 0.25 microampere. The internal resistance of the microammeter shall be between 50 and 150 ohms.

Source of Current

5. A 6-v. automobile starting and lighting storage battery or a source of constant emf. shall be used for supplying current to the lamp.

Sieve

6. The sieve shall be 2 in. in diameter and have side walls 3 in. in height, and shall be fitted with a No. 325 (44-micron) woven wire screen cloth, which cloth shall conform to the requirements of the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials,[4] and be such that the sieve correction factor obtained shall not be greater than 15 per cent (see Section 10 (*b*)).

Stirring Apparatus

7. The stirring apparatus shall consist of either (*a*) a cylindrical brush, $\frac{3}{4}$ in. in diameter and about $1\frac{3}{4}$ in. in length, with an end approximately fitting the contour of the bottom of a $\frac{7}{8}$-in. diameter test tube, or (*b*) any other stirring device that will be equally efficient in dispersion as measured by specific surface determinations on a standard sample. The stirring apparatus shall rotate at a speed of approximately 3500 rpm.

Timing Burette

8. The time of settling for the different sized particles shall be obtained from a burette from which kerosine is allowed to flow. The burette shall consist of a glass tube having a capillary tube fused into the lower end. The upper end of the large tube shall be flared to serve as a funnel for introducing kerosine into the tube. The burette shall conform to the following limiting dimensions:[a]

	Dimension, cm.	Permissible Variation, cm.
Length of large tube	38	± 4
Inside diameter of large tube	1.9	± 0.2
Length of capillary	17.5	± 2.5
Diameter of capillary	0.09	± 0.005
Top of burette to zero line	7	± 1

[a] Since glass tubing of desired dimensions is not always obtainable, the above wide permissible variations allow selection of dimensions to produce a burette having a duration of flow which will permit of calibration as described in Section 10 (*a*).

Suspending Liquid

9. Clear white kerosine shall be used with the turbidimeter apparatus.

CALIBRATION OF APPARATUS

Calibration of Turbidimeter

10. The turbidimeter apparatus shall be calibrated in accordance with the following procedure:

(*a*) *Calibration of Burette Scale.*—For calibration of the burette scale a kerosine having a known viscosity and density for the temperature at which the calibration is to be made shall be used. The times of flow from the burette which correspond to the times of settling for the different sized particles shall be calculated from the following equation:

$$t = \frac{1{,}837{,}000\,u}{(P_1 - P_2)} \times \frac{h}{d^2}$$

where:

t = time of settling, or time of flow in seconds,
u = viscosity of kerosine in poises,
P_1 = density of cement particles in grams per cubic centimeter = 3.15 for portland cement,[a]

[4] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

[a] The specific gravity of portland cement does not vary greatly, and in this work it is considered constant at 3.15. A variation of 0.15 from this value when substituted in Stokes' law gives a variation of 2.5 per cent in the diameter of the particle measured.

P_2 = density of kerosine in grams per cubic centimeter,
h = depth of suspension to level of light in centimeters, and
d = diameter of particle in microns.

Values of $\frac{h}{d^2}$ are given in Table I.

The burette shall next be filled with kerosine at the calibrating temperature, a timing clock shall be started at the instant the kerosine in the burette drains past the zero line, and levels reached by the draining kerosine shall be marked on the burette for each of the time intervals, t, calculated as described above. At these marks, permanent lines and numbers indicating the corresponding diameters shall be etched on the burette (Note 1).

The construction and graduation of the burette shall be such that the time required for the kerosine to pass the permanent lines of the burette shall agree with the calculated time of settling within 1 per cent, except that the permissible variation shall be not less than 1 sec.

NOTE 1.—By using the calibrated burette the apparatus may be used within the normal range of room temperatures without further correction, the change in rate of flow of the kerosine from the burette automatically compensating for change in viscosity of the suspension due to temperature. The kerosine in the burette and that of the suspension should be kept the same within 1 F. (0.5 C.). This condition will ordinarily exist if the supply of kerosine is kept in the same room as the apparatus.

Extreme care must be taken to ascertain that only clean kerosine is used in the burette, and in addition the capillary should be examined frequently to make sure that no small pieces of lint or other foreign material have become lodged in it.

(*b*) *Calibration of No. 325 Sieve.*—The spray nozzle and pressure gage shall be connected to a water supply line and the pressure on the spray nozzle adjusted to 10 psi. One gram of the current lot of National Bureau of Standards' Standard Sample No. 114[a] shall be placed on the No. 325 (44-micron) sieve and washed for 1 min. The sieve and residue shall be dried in an oven or over a hot plate (Note 2) and the residue brushed from the sieve and weighed on an analytical balance. The difference between the amount of residue obtained in this manner and the amount of residue indicated by the fineness specified for the standard sample, expressed as a percentage of the former residue, shall be the sieve correction factor (see Note 3; also Table II, footnote *a*).

NOTE 2.—Care should be taken not to heat the sieve hot enough to soften the solder.

NOTE 3.—It should be observed that the sieve correction as specified is a *factor* to be *multiplied* by the residue obtained and that the amount to be added to or subtracted from the residue in any given test is therefore proportional to the amount of residue.

(*c*) *Determination of the Proper Light Intensity, I_0.*—The calibration of the turbidimeter proper shall be made by adjustment of the light intensity, I_0. Increasing the light intensity reduces the calculated surface area of any sample and *vice versa*. Using the regular test procedure described in Sections 12 to 14, and with the light adjusted to a trial value of 25 microamperes through the filter, a determination of the specific surface shall be made using the current lot of National Bureau of Standards' Standard Sample No. 114.[a] If a specific surface greater than that assigned for the standard sample is obtained, the light intensity through the filter shall be increased, the suspension shall be reagitated, and the analysis repeated. If a specific surface less than the assigned value is obtained, the light intensity shall be decreased, the suspension reagitated, and the analysis repeated. Using

[a] Obtainable at the National Bureau of Standards Washington, D. C.

new suspensions, the above procedure shall be repeated twice and the average of the three intensities noted. When the intensity is found which results in a specific surface within plus or minus 15 sq. cm. of the values assigned to the standard sample, the tank shall be removed and the intensity through the filter recorded. The light shall next be adjusted to this average value and a tank of clear kerosine placed in its path. The intensity through the filter plus tank of clear kerosine shall be recorded. This becomes the permanent reference value for the intensity of the light, and the rheostats shall be adjusted at the beginning of each test to give this value through the filter plus tank of clear kerosine from the regular supply.

TEST SAMPLE

Size of Test Sample

11. The size of the sample of cement for test shall be so selected that the microammeter readings will occur in the middle portion of the microammeter scale (Note).

NOTE.—The following approximations will be helpful in many instances in selecting the size of sample: 0.3-g. sample for a cement of which more than 85 per cent passes a No. 325 (44-micron) sieve, 0.4-g. sample for a cement of which 70 to 85 per cent passes a No. 325 sieve, and 0.5-g. sample for a cement of which less than 70 per cent passes a No. 325 sieve.

PROCEDURE

Sieve Determination

12. A No. 325 (44-micron) sieve determination shall be made on a 1-g. sample of the cement in accordance with the procedure specified for calibration in Section 10 (*b*). The percentage (corrected) of cement passing the sieve shall be recorded as *r*. (See Note; also Table II, footnote *a*.)

NOTE.—After three determinations the sieve should be dipped in diluted HCl (1:10) and immediately rinsed with water to remove particles lodged in the meshes. After 25 determinations the sieve should be recalibrated.

Preparation of the Suspension

13. (*a*) The cement sample shall be placed in a test tube with 10 to 15 ml. of kerosine and 5 drops of oleic acid (U.S.P.). (Oleic acid is added as a dispersing agent and has slight or no effect on the viscosity of the kerosine. The oleic acid, which changes with age and exposure to heat and light, should be kept in a well-stoppered dark-glass bottle.) The mixture shall then be stirred for 1 min. with the apparatus described in Section 7 and shall then be transferred to the settling tank, the brush and tube washed with clear kerosine which shall also be added to the suspension, and clear kerosine added until the total volume of the suspension in the tank is 335 ml. (Note 1).

NOTE 1.—Since it is important that the level of the liquid in the tank coincide with the zero mark on the scale of the instrument (see Section 3 (*g*)), it is correspondingly important that the volume of the suspension be close to 335 ml., as specified. This may be accomplished by the use of a flask, calibrated to deliver 335 ml., from which all the kerosine used in making up a suspension is taken; or a point gage may be used, consisting essentially of a flat plate approximately 2 by 2½ in. to the center of which is attached vertically a metal pin of such length that, when the plate is placed in position on top of the tank, the point of the pin is at the 335-ml. level. Either of these methods should insure the position of the liquid level within a few tenths of a millimeter.

(*b*) *Agitation of the Suspension.*—Just before being placed in the path of the light beam, the tank shall be agitated to effect a uniformity of the suspension. The tank shall be covered with a ground-glass cover and oscillated 180 deg. about a horizontal axis, turning the tank upside down and back again to its original upright position approximately once each

second for about 1 min. (Note 2). Care should be taken to prevent loss of kerosine (Note 3). The suspension will then be ready to place in its proper position in the path of the light beam.

NOTE 2.—The procedure described for oscillating the suspension must be carefully followed. The procedure used in shaking a cocktail must not be used.

NOTE 3.—The faces of the glass tank shall be clean when the suspension is added, and care must be taken to avoid as much as possible the necessity of cleaning the faces thereafter.

Procedure

14. The turbidimeter apparatus shall be operated in accordance with the procedure described in the following Paragraphs (*a*) to (*h*):

(*a*) With retarding filter and sedimentation tank containing approximately 100 ml. of clear kerosine in the light path, the light shall be adjusted to the proper intensity, I_0, by taking repeated readings at 1-min. intervals until an unchanging value shall indicate that the lamp and photoelectric cell are in equilibrium (Note 1).

NOTE 1.—To protect the microammeter, the lamp must never be turned on without a suspension or the retarding filter in position to reduce the light intensity to a value within the range of the microammeter.

(*b*) The proper size of sample of cement (Section 11) shall be weighed and a suspension prepared in accordance with Section 13 (*a*).

(*c*) The shelf level pointer shall be placed at the 30-60-micron position.

(*d*) The burette shall be filled to a suitable height with kerosine from the same lot and at the same temperature as the kerosine used in the suspension, and oscillation of the tank containing the suspension shall be started in accordance with Section 13 (*b*). The oscillation shall be continued until the kerosine drains to the zero line on the burette, when the agitation shall be stopped (Note 2). The tank shall be immediately placed in position in the path of the light beam.

NOTE 2.—The lid to the tank must fit so that no kerosine drains down the outside faces of the tank.

(*e*) The retarding filter shall be immediately removed from the light path and the cabinet door closed.

(*f*) The microammeter shall be read at the instants the kerosine in the burette drains past marks 60, 55, 50, 45, 40, 35, and 30.

(*g*) The shelf shall then be raised successively to the marks 25, 20, 15, 10, and 7.5 on the pointer scale, the microammeter being read at each position as the kerosine drains past the corresponding mark on the burette.

(*h*) The filter shall be replaced in the path of the light beam, the tank removed, and the intensity of the lamp checked (Note 3).

NOTE 3.—If the microammeter indication has shifted more than 0.3 microampere from the initial setting through the filter alone, the test must be repeated. A freshly charged storage battery should be momentarily short-circuited to reduce the voltage to constant value. If the microammeter indication continues to fluctuate, look for loose connections in the lamp and photoelectric-cell circuits, see that the rheostat contacts are clean, and make certain that the lamp, socket, and reflector are rigidly fixed in their mounting.

CALCULATION

Calculation and Record

15. (*a*) *Specific Surface.*—Specific surface shall be calculated from the following equation:

$$S = \frac{38r(2 - \log I_{60})}{1.5 + 0.75 \log I_{7.5} + \log I_{10} + \log I_{15} + \log I_{20} \ldots + \log I_{55} - 11.5 \log I_{60}}$$

where:

S = specific surface of the sample in square centimeters per gram,

r = corrected percentage by weight passing the No. 325 (44-micron) sieve, and

$I_{7.5}$, I_{10}, I_{15} ... I_{60} = microammeter readings, in microamperes, which correspond to the particle diameters 7.5, 10, 15 ... 60 microns.

NOTE.—The constant factor 38 applies only to a material having the specific gravity of portland cement (approximately 3.15). For any other material there must be calculated the corresponding proper value for this factor which, in the derivation of the formula, varies inversely as the density of the particles (in grams per cubic centimeter).

(*b*) *Recording of Data.*—The form shown in Table II is suggested for the recording of turbidimeter data and calculation of specific surface.

Calculation of Specific Surface from First Turbidity Reading I_{60}

16. (*a*) This method may be used for successive determinations from the same plant, provided, that the same size test samples are used, and that there is no great change in fineness, color, or other properties of the cement. Under these conditions the specific surface of a sample may be calculated from the first turbidity reading, I_{60}, by using the following equation:

$$S = c(2 - \log I_{60})$$

where:

S = specific surface of the sample in square centimeters per gram,

I_{60} = microammeter reading corresponding to particle diameter of 60 microns, and

c = transmittancy constant for the particular plant.

NOTE.—The transmittancy constant "c" may be evaluated by substituting in the above equation known values of S and $2 - \log I_{60}$ as determined from a complete turbidimeter test. The average of not less than five values of "c," as determined from complete tests, should be used in the above equation.

(*b*) If the fineness of a sample as determined in accordance with Paragraph (*a*) fails to conform to the specification requirements for fineness, a retest shall be made by the complete procedure described in Sections 14 and 15.

TABLE II.—ILLUSTRATIVE FORM FOR RECORDING TURBIDIMETER TEST DATA AND CALCULATION OF SPECIFIC SURFACE.

(Values shown are for purpose of illustration only.)

Sample Identification	Sample X
Passing No. 325 (44-micron) Sieve (wet method), r, corrected	89.7 per cent[a]
Filter Reference (through filter alone)	
Before Test	20.5 microamperes
After Test	20.5 microamperes
Weight of Sample Tested	0.3 g.

Particle Size, microns	I, microamperes		Log I
60	11.0	.....	1.041[b]
55	11.1	1.045	
50	11.3	1.053	
45	11.5	1.061	
40	11.7	1.068	
35	12.1	1.083	
30	12.6	1.100	
25	13.4	1.127	
20	14.4	1.158	
15	15.7	1.196	
10	19.1	1.281	
7.5	23.0	.....	1.362[b]
	0.75×1.362 =	1.022	
		1.500	
	Sum =	13.694	
	11.5×1.041 =	11.972	
	Difference =	1.722	

$$S = \frac{38 \times 89.7 \times 0.959}{1.722} = 1900 \text{ sq. cm. per g.}$$

[a] The following example presents an illustration of the determination and application of the sieve correction factor for a No. 325 sieve (wet method):

Determination:

Residue specified for standard sample No. 114c = 10.8 per cent, or a residue for 1-g. test sample	= 0.108 g.
Residue obtained on the sieve, which is being calibrated with standard sample	= 0.100 g.
Difference between specified and test residues	= 0.008 g.
Correction factor for sieve (plus in this example)	$= +\frac{0.008}{0.100} \times 100 = +8.0$ per cent

Application:

Sieve correction factor	= + 8.0 per cent
Residue from the sample under test	= 0.095 g.
Correction = + 0.08 × 0.095 g.	= +0.008 g.
Corrected residue (algebraic sum)	= 0.103 g. = 10.3 per cent
Corrected amount passing sieve (r)	= 89.7 per cent

[b] For convenience in calculation, log I_{60} and log $I_{7.5}$ are recorded in a separate column.

Standard Method of Test for

FINENESS OF HYDRAULIC CEMENT BY THE NO. 200 SIEVE[1]

A.S.T.M. Designation: C 184 – 44

ADOPTED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 184; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test is intended for determining the fineness of hydraulic cement by means of the No. 200 (74-micron) sieve.

Apparatus

2. (*a*) *Sieve.*—A standard No. 200 (74-micron) sieve shall be used, conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11)[3] and certified by the National Bureau of Standards. Wire cloth for standard sieves for cement shall be woven (not twilled) from brass, bronze, or other suitable wire, and mounted without distortion on a frame, about 2 in. (5 cm.) below the top of the frame. The joint between the cloth and frame shall be smoothly filled with solder to prevent lodging of the cement. The sieve frame shall be circular, 8 in. (20.32 cm.) in diameter, and shall be provided with a pan and cover. The correction for the sieve used in this test shall be determined by sieving tests, made in conformity with the procedure of this test method, using a cement standardized for this purpose by the National Bureau of Standards (standard sample No. 46 or No. 47). The difference between the percentage residue on the sieve and that assigned to the standard sample is the amount of the correction, which shall be added or subtracted as necessary.

(*b*) *Balance.*—On balances in use, the permissible variation at a load of 50 g. shall be plus or minus 0.05 g., and at loads less than 0.1 g. the permissible variation shall be plus or minus 0.01 g. The permissible variations on new balances shall be one half of these values. The sensibility reciprocal[4] at each of the

[1] Under the standardization procedure of the Society this method is under the jurisdiction of the A.S.T.M. Committee C-1 on Cement.

[2] This method formerly constituted a portion of the Standard Methods of Sampling and Physical Testing of Portland Cement (C 77 – 40), discontinued in 1944, which latter standard was originally part of the Standard Specifications for Portland Cement (A.S.T.M. Designation: C 9) published as standard from 1904 to 1930, at which time the specifications and methods were separated and published individually.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

[4] The sensibility reciprocal is a measure of the sensitiveness of a balance, and is the weight required to move the position of equilibrium of the pointer one division. For a complete definition of sensibility reciprocal, see "Specifications, Tolerances, and Regulations for Commercial Weighing and Measuring Devices," *Handbook H29*, Nat. Bureau Standards, September, 1942, pp. 87 and 88.

specified loads shall be not greater than twice the permissible variation specified for the load in question. The balance shall be enclosed in a glass case.

(*c*) *Weights.*—The permissible variations on weights in use in the fineness test shall be as shown in Table I, and the permissible variations on new weights shall be one half of these values.

TABLE I.—PERMISSIBLE VARIATIONS ON WEIGHTS.

Weight, g.	Permissible Variations on Weights in Use, plus or minus, g.
50	0.04
20	0.02
10	0.014
5	0.010
2	0.006
1	0.004
0.500	0.003
0.200	0.002
0.100	0.001
0.050	0.001
0.020	0.001
0.010	0.001

(*d*) *Brush.*—A bristle brush will be required for use in cleaning the No. 200 sieve. A 1- or 1½-in. brush with a 10-in. handle is a convenient size.

Procedure

3. (*a*) A 50-g. sample of the cement shall be placed on the clean, dry No. 200 (74-micron) sieve with the pan attached. Washers, shot, and slugs shall not be used on the sieve. While holding the sieve and pan (without cover) in both hands, the sieving shall be done with a gentle wrist motion until most of the fine material has passed through and the residue looks fairly clean. This operation usually requires only 3 or 4 min. When the residue appears clean the cover shall be placed on the sieve and the pan removed. Then, with the sieve and cover held firmly in one hand, the side of the sieve shall be tapped gently with the handle of the brush used for cleaning the sieve. Dust adhering to the sieve will thus be dislodged and the underside of the sieve may then be swept clean. The pan shall be emptied and wiped out thoroughly with a cloth or waste, the sieve replaced in the pan, and the cover carefully removed. Any coarser material that has been caught in the cover during the tapping shall then be returned to the sieve.

(*b*) The sieving shall be continued (with the cover removed) as described in Paragraph (*a*) for 5 or 10 min., depending on the condition of the cement. The gentle wrist motion involves no danger of spilling the residue, which shall be kept well spread out on the screen. More or less continuous rotation of the sieve shall be carried on throughout the sieving. This open sieving may usually be continued safely for 8 min. or more, but care should be taken that it is not continued too long. The cover shall then be replaced and the same process of cleaning followed as described in Paragraph (*a*). If the cement is in proper condition, there should now be no appreciable dust remaining in the residue nor adhering to sieve or pan.

(*c*) One-minute tests shall be made as follows: The sieve, with pan and cover attached, shall be held in one hand in a slightly inclined position and moved forward and backward in the plane of inclination, at the same time striking the side gently about 150 times per minute against the palm of the other hand on the upstroke. The sieving shall be performed over a white paper, and any material escaping from the sieve or pan and collected on the paper shall be returned to the sieve. After every 25 strokes, the sieve shall be turned about one sixth of a revolution, in the same direction (Note). The sieving operation shall be continued until not more than 0.05 g. passes through in 1 min. of continuous sieving. Then the residue on the sieve shall be transferred to the

balance pan, taking care to brush the sieve cloth thoroughly from both sides to insure the removal of all the residue from the sieve.

NOTE.—Sieve covers may be marked with three straight lines through the center and intersecting at 60 deg. If one of the lines is marked with an arrowhead and the habit is formed of starting with this point under the right hand, one can easily follow the progress of the 1-min. tests.

Calculation

4. The fineness shall be calculated as follows:

$$F = \frac{R}{50} \times 100 + C$$

where:

F = fineness of the cement expressed as percentage residue on the No. 200 (74-micron) sieve,

R = weight of residue on the No. 200 (74-micron) sieve, in grams, and

C = sieve correction, determined as prescribed in Section 2 (*a*) and added as an algebraic sum.

Mechanical Sieving

5. Mechanical sieving devices may be used, but the cement shall not be rejected if it meets the fineness requirement when tested by the hand method described in Section 3.

Standard Method of Test for

NORMAL CONSISTENCY OF HYDRAULIC CEMENT[1]

A.S.T.M. Designation: C 187 - 44

ADOPTED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 187; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test is intended for determining the normal consistency of hydraulic cement.

Apparatus

2. (*a*) *Scales.*—The scales shall conform to the following requirements: On scales in use the permissible variation at a load of 1000 g. shall be plus or minus 1.0 g. The permissible variation on new scales shall be one half of this value. The sensibility reciprocal[3] shall be not greater than twice the permissible variation.

(*b*) *Weights.*—The permissible variations on weights in use in weighing the cement shall be as prescribed in Table I. The permissible variations on new weights shall be one half of the values in Table I.

(*c*) *Glass Graduates.*—Glass graduates of 100-ml. to 200-ml. capacities used for measuring the mixing water shall be made to deliver the indicated volume at 20 C. (68 F.). The permissible variation shall be plus or minus 1.0 ml. The main graduation lines shall be circles and shall be numbered. The least graduations shall extend at least one seventh of the way around, and intermediate graduations shall extend at least one fifth of the way around.

TABLE I.—PERMISSIBLE VARIATIONS ON WEIGHTS.

Weight, g.	Permissible Variations on Weights in Use, plus or minus, g.
500	0.35
300	0.3
250	0.25
200	0.20
100	0.15
50	0.10
20	0.05
10	0.04
5	0.03
2	0.02
1	0.01

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-1 on Cement.

[2] This method formerly constituted a portion of the Standard Methods of Sampling and Physical Testing of Portland Cement (C 77 - 40), discontinued in 1944, which latter standard was originally part of the Standard Specifications for Portland Cement (A.S.T.M. Designation: C 9) published as standard from 1904 to 1930, at which time the specifications and methods were separated and published individually.

[3] The sensibility reciprocal is a measure of the sensitiveness of a balance, and is the weight required to move the position of equilibrium of the pointer one division. For a complete definition of sensibility reciprocal, see "Specifications, Tolerances, and Regulations for Commercial Weighing and Measuring Devices," *Handbook H29*, Nat. Bureau Standards, September, 1942, pp. 87 and 88.

(*d*) *Vicat Apparatus.*—The Vicat apparatus shall consist of a frame *A* (Fig. 1) bearing a movable rod *B*, weighing 300 g., one end *C*, the plunger end, being 1 cm. in diameter for a distance of at least 5 cm., and the other end having a removable needle *D*, 1 mm. in diameter and 5 cm. in length. The rod *B* is re-

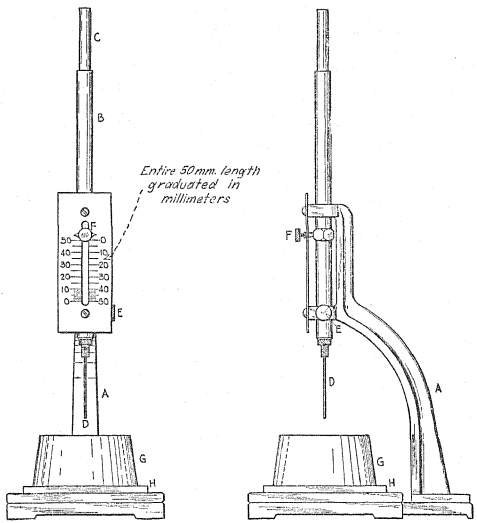


FIG. 1.—Vicat Apparatus.

versible, and can be held in any desired position by a set screw *E*, and has an adjustable indicator *F* which moves over a scale (graduated in millimeters) attached to the frame *A*. The paste is held in a rigid conical ring *G*, resting on a glass plate *H* about 10 cm. square. The ring shall be made of a noncorroding, nonabsorbent material, and shall have an inside diameter of 7 cm. at the base and 6 cm. at the top, and a height of 4 cm. In addition to the above, the Vicat apparatus shall conform to the following requirements:

Weight of plunger	300 ± 0.5 g. (0.661 lb. ± 8 grains)
Diameter of larger end of plunger	1 ± 0.005 cm. (0.394 ± 0.002 in.)
Diameter of needle	1 ± 0.05 mm. (0.039 ± 0.002 in.)
Inside diameter of ring at bottom	7 ± 0.3 cm. (2.75 ± 0.12 in.)
Inside diameter of ring at top	6 ± 0.3 cm. (2.36 ± 0.12 in.)
Height of ring	4 ± 0.1 cm. (1.57 ± 0.04 in.)
Graduated scale	The graduated scale, when compared with a standard scale accurate to within 0.1 mm. at all points, shall not show a deviation at any point greater than 0.25 mm.

Temperature

3. The temperature of the air in the vicinity of the mixing slab, the dry ce-

ment, molds, and base plates shall be maintained between 20 and 27.5 C. (68 and 81.5 F.). The temperature of the mixing water shall not vary from 21 C. (70 F.) by more than plus or minus 1.7 C. (3 F.).

Procedure.

4. (*a*) *Preparation of Cement Paste.*—A 500-g. sample of cement shall be placed on a smooth, nonabsorbent surface and a crater formed in the center. A measured quantity of clean water shall be poured into the crater, and the cement on the outer edge shall be turned into the crater within 30 sec. by the aid of a trowel. After an additional interval of 30 sec. for the absorption of the water, during which interval the dry cement around the outside of the cone shall be lightly troweled over the remaining mixture to reduce the evaporation losses and to promote absorption, the operation shall be completed by continuous, vigorous mixing, squeezing, and kneading with the hands for 1½ min. During the operation of mixing, the hands shall be protected by snug-fitting rubber gloves.

(*b*) *Molding Test Specimen.*—The cement paste, prepared as described in Paragraph (*a*), shall be quickly formed into a ball with the gloved hands and tossed six times from one hand to the other, maintaining the hands about 6 in. apart. The ball, resting in the palm of one hand, shall be pressed into the larger end of the conical ring *G*, Fig. 1, held in the other hand, completely filling the ring with paste. The excess at the larger end shall then be removed by a single movement of the palm of the hand. The ring shall then be placed on its larger end on a glass plate *H*, and the excess paste at the smaller end sliced off at the top of the ring by a single oblique stroke of a sharp-edged trowel held at a slight angle with the top of the ring, and the top smoothed, if necessary, with a few light touches of the pointed end of the trowel. During these operations of cutting and smoothing, care shall be taken not to compress the paste.

(*c*) *Consistency Determination.*—The paste confined in the ring, resting on the plate, shall be centered under the rod *B*, Fig. 1, the plunger end *C* of which shall be brought in contact with the surface of the paste, and the setscrew *E* tightened. Then the movable indicator *F* shall be set to the upper zero mark of the scale, or an initial reading taken, and the rod released 30 sec. after completion of mixing. The apparatus shall be free of all vibrations during the test. The paste shall be of normal consistency when the rod settles to a point 10 mm. below the original surface in 30 sec. after being released. Trial pastes shall be made with varying percentages of water until the normal consistency is obtained. Each trial shall be made with fresh cement.

Calculation

5. The amount of water required for normal consistency shall be calculated as a percentage by weight of the dry cement.

Standard Method of Test for

SOUNDNESS OF HYDRAULIC CEMENT OVER BOILING WATER (PAT TEST)[1]

A.S.T.M. Designation: C 189 – 44

ADOPTED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 189; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test is intended for determining the soundness of hydraulic cement by means of neat cement pats in steam at atmospheric pressure.

Apparatus

2. A steam apparatus in which the pats can be maintained in an atmosphere of saturated steam above boiling water, similar to that shown in Fig. 1, is recommended. The capacity of this apparatus may be increased by using a rack for holding the pats in a vertical or inclined position.

Test Specimen

3. A pat from cement paste of normal consistency,[3] about 3 in. in diameter, $\frac{1}{2}$ in. in thickness at the center, and tapering to a thin edge, shall be made on a flat, clean glass plate about 4 in. square. In molding the pat, the cement paste shall first be flattened on the glass and the pat then formed by drawing the trowel from the outer edge toward the center to give a rounded surface.

Procedure

4. (*a*) Immediately after being made, the pats shall be stored for 24 hr. in an atmosphere of not less than 98 per cent relative humidity at 70 ± 3 F. The pat on the glass plate shall then be placed in an atmosphere of saturated steam on a suitable support 1 in. above boiling water for 5 hr.

(*b*) The pat shall be examined for soundness within 1 hr. after removal from the steam apparatus. Unsoundness is usually manifested by change in volume, which causes distortion, cracking, checking, or disintegration as illustrated in Fig. 2 (*b*).

(*c*) Should the pat leave the plate, distortion may be detected best with a straightedge applied to the surface that was in contact with the plate.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-1 on Cement.

[2] This method formerly constituted a portion of the Standard Methods of Sampling and Physical Testing of Portland Cement (C 77 – 40), discontinued in 1944, which latter standard was originally part of the Standard Specifications for Portland Cement (A.S.T.M. Designation: C 9) published as standard from 1904 to 1930, at which time the specifications and methods were separated and published individually.

[3] Standard Method of Test for Normal Consistency of Hydraulic Cement (A.S.T.M. Designation: C 187), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

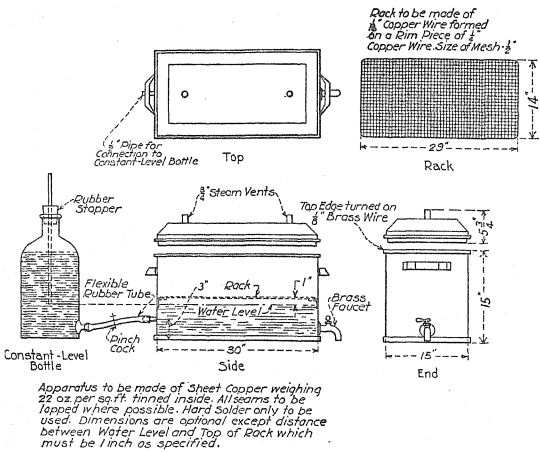
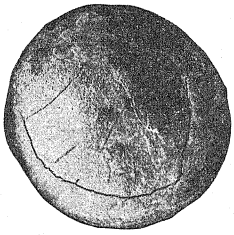
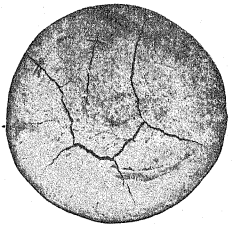


FIG. 1.—Apparatus for Pat Test for Soundness.

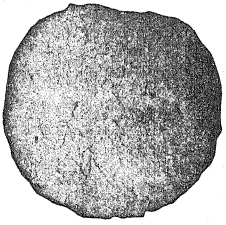




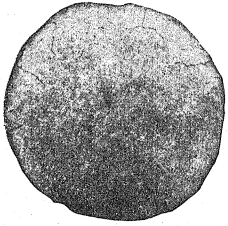
(*a*) Shrinkage Cracks Shown Above, Due to Exposure of Pats to Dry Air During Setting, Are Not to Be Considered as Evidence of Unsoundness.



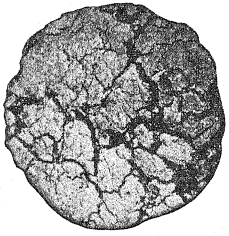
Distortion



Checking



Cracking



Disintegration

(*b*) Typical Failures in Soundness Test.

FIG. 2.—Soundness Test Pats.

Standard Method of Test for

SPECIFIC GRAVITY OF HYDRAULIC CEMENT[1]

A.S.T.M. Designation: C 188 – 44

ADOPTED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 188; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test is intended for determining the specific gravity of hydraulic cement. Its particular usefulness is in connection with the design and control of concrete mixtures.

Apparatus

2. (*a*) The standard Le Chatelier flask[3] conforming to the dimensions shown in Fig. 1 shall be used.

(*b*) Kerosine free of water, or naphtha having a gravity not lighter than 62° A.P.I. shall be used in the specific gravity determination.

Procedure

3. (*a*) Specific gravity of cement shall be determined on the material as received, unless otherwise specified. If the specific gravity determination on a loss-free sample is required, the sample shall first be ignited as described in the test for loss on ignition in Section 17 of the Standard Methods of Chemical Analysis of Portland Cement (A.S.T.M. Designation: C 114).[4]

(*b*) The flask shall be filled (Note 1) with either of the liquids specified in Section 2 (*b*) to a point on the stem between the zero and the 1-ml. mark. The inside of the flask above the level of the liquid shall be dried, if necessary, after pouring. The first reading shall be recorded after the flask has been immersed in the water bath (Note 2) in accordance with Paragraph (*c*). A weighed quantity of cement (about 64 g. for portland cement) shall then be introduced in small amounts at the same temperature as the liquid (Note 1). Care shall be taken to avoid splashing and to see that the cement does not adhere to the inside of the flask above the liquid. A vibrating apparatus may be used to accelerate the introduction of the cement into the flask

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-1 on Cement.

[2] This method formerly constituted a portion of the Standard Methods of Sampling and Physical Testing of Portland Cement (C 77 – 40), discontinued in 1944, which latter standard was originally part of the Standard Specifications for Portland Cement (A.S.T.M. Designation: C 9) published as standard from 1904 to 1930, at which time the specifications and methods were separated and published individually.

[3] This apparatus has been standardized by the National Bureau of Standards. Requirements for the flask are described in "Testing of Glass Volumetric Apparatus," Nat. Bureau Standards, *Circular C434*, Superintendent of Documents, Government Printing Office, Washington, D. C. (1941).

[4] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

and to prevent the cement from sticking to the neck. After all the cement has been introduced, the stopper shall be placed in the flask and the flask rolled in an inclined position (Note 1), or gently whirled in a horizontal circle, so as to free the cement from air until no further air bubbles rise to the surface of the liquid. If a proper amount of cement has been added, the level of the liquid will be in its final position at some point of the upper series of graduations. The final reading shall be taken after the flask has been immersed in the water bath in accordance with Paragraph (*c*).

NOTE 1.—It is advisable to use a rubber pad on the table top when filling or rolling the flask.

NOTE 2.—Before the cement has been added to the flask, a loose-fitting lead-ring weight around the stem of the flask will be helpful in holding the flask in an upright position in the water bath, or the flask may be held in the water bath by a burette clamp.

(*c*) The flask shall be immersed in a constant-temperature water bath, maintained at about room temperature, for a sufficient interval before making either of the readings so as to avoid variations greater than 0.2 C. in the temperature of the liquid in the flask. All readings shall be checked until they are constant to ensure that the contents of the flask have reached the temperature of the water bath.

Calculation

4. The difference between the first and the final readings represents the volume of liquid displaced by the weight of cement used in the test. The specific gravity shall be calculated as follows:

$$\text{Sp. gr.} = \frac{\text{wt. of cement in grams}}{\text{displaced vol. in ml.}}$$

Reproducibility

5. Duplicate determinations of specific gravity by this method should agree within 0.01.

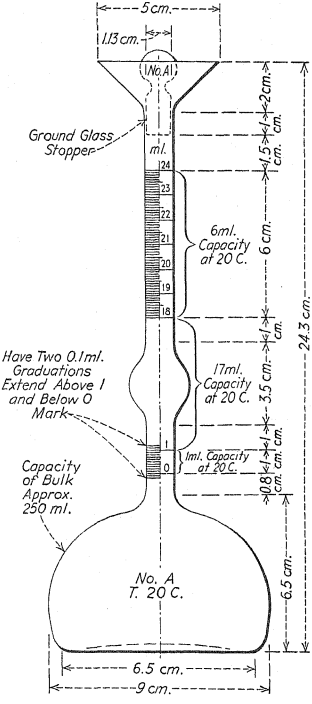


FIG. 1.—Le Chatelier Flask for Specific Gravity Test.

NOTE.—Variations of a few millimeters in such dimensions as total height of flask, diameter of base, etc., are to be expected and will not be considered sufficient cause for rejection.

Standard Methods of Test for

TIME OF SETTING OF HYDRAULIC CEMENT BY THE VICAT OR GILLMORE NEEDLES[1]

A.S.T.M. Designation: C 191 – 44

ADOPTED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 191; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods of test are intended for determining the time of setting of hydraulic cement by means of the Vicat or Gillmore needles.

NOTE.—The Vicat method and the Gillmore method are alternate procedures, either of which may be used as specified.

Apparatus

2. (*a*) *Scales.*—The scales shall conform to the following requirements: On scales in use the permissible variation at a load of 1000 g. shall be plus or minus 1.0 g. The permissible variation on new scales shall be one half of this value. The sensibility reciprocal[3] shall be not greater than twice the permissible variation.

(*b*) *Weights.*—The permissible variations on weights in use in weighing the cement shall be as prescribed in Table I. The permissible variations on new weights shall be one half of the values in Table I.

TABLE I.—PERMISSIBLE VARIATIONS ON WEIGHTS.

Weight, g.	Permissible Variations on Weights in Use, plus or minus, g.
500	0.35
300	0.3
250	0.25
200	0.20
100	0.15
50	0.10
20	0.05
10	0.04
5	0.03
2	0.02
1	0.01

(*c*) *Glass Graduates.*—Glass graduates of 100-ml. to 200-ml. capacities used for measuring the mixing water shall be made to deliver the indicated volume at 20 C. (68 F.). The permissible variation shall be plus or minus 1.0 ml. The main graduation lines shall be circles and shall be numbered. The least graduations shall extend at least one seventh of the

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee C-1 on Cement.

[2] These methods formerly constituted a portion of the Standard Methods of Sampling and Physical Testing of Portland Cement (C 77 – 40), discontinued in 1944, which latter standard was originally part of the Standard Specifications for Portland Cement (A.S.T.M. Designation: C 9) published as standard from 1904 to 1930, at which time the specifications and methods were separated and published individually.

[3] The sensibility reciprocal is a measure of the sensitiveness of a balance, and is the weight required to move the position of equilibrium of the pointer one division. For a complete definition of sensibility reciprocal, see "Specifications, Tolerances, and Regulations for Commercial Weighing and Measuring Devices," *Handbook H29*, Nat. Bureau Standards, September, 1942, pp. 87 and 88.

way around, and intermediate graduations shall extend at least one fifth of the way around.

(*d*) *Vicat Apparatus.*—The Vicat apparatus shall consist of a frame *A* (Fig. 1) bearing a movable rod *B*, weighing 300 g., one end *C*, the plunger end, being 1 cm. in diameter for a distance of at least 5 cm., and the other end having a removable needle *D*, 1 mm. in diameter and 5 cm. in length. The rod *B* is reversible, and can be held in any desired position by a set screw *E*, and has an adjustable indicator *F* which moves over a scale (graduated in millimeters) attached to the frame *A*. The paste is held in a rigid conical ring *G*, resting on a glass plate *H* about 10 cm. square. The ring shall be made of a noncorroding, nonabsorbent material, and shall have an inside diameter of 7 cm. at the base and 6 cm. at the top, and a height of 4 cm. In addition to the above, the Vicat apparatus shall conform to the following requirements:

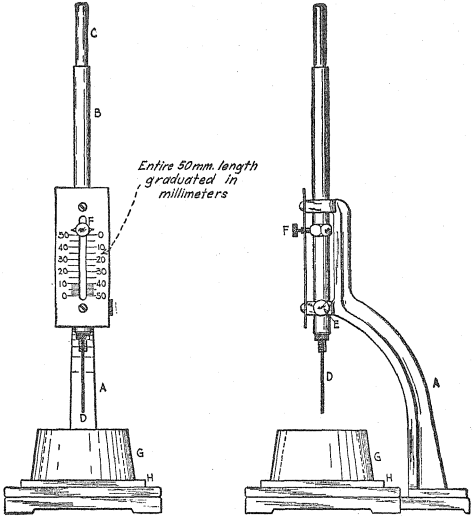


FIG. 1.—Vicat Apparatus.

Weight of plunger	300 ± 0.5 g. (0.661 lb. ± 8 grains)
Diameter of larger end of plunger	1 ± 0.005 cm. (0.394 ± 0.002 in.)
Diameter of needle	1 ± 0.05 mm. (0.039 ± 0.002 in.)
Inside diameter of ring at bottom	7 ± 0.3 cm. (2.75 ± 0.12 in.)
Inside diameter of ring at top	6 ± 0.3 cm. (2.36 ± 0.12 in.)
Height of ring	4 ± 0.1 cm. (1.57 ± 0.04 in.)
Graduated scale	The graduated scale, when compared with a standard scale accurate to within 0.1 mm. at all points, shall not show a deviation at any point greater than 0.25 mm.

(*e*) *Gillmore Needles.*—The Gillmore needles shall conform to the following requirements:

Initial Needle:	
Weight	¼ lb. ± 8 grains (113.4 ± 0.5 g.)
Diameter	1/12 ± 0.002 in. (2.12 ± 0.05 mm.)
Final Needle:	
Weight	1 lb. ± 8 grains (453.6 ± 0.5 g.)
Diameter	1/24 ± 0.002 in. (1.06 ± 0.05 mm.)

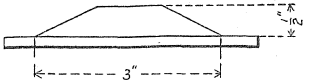
The needle tips shall be cylindrical for a distance of about 1/16 in. The needle ends shall be plane and at right angles to the axis of the rod and shall be maintained in a clean condition. The Gillmore needles should preferably be mounted as shown in Fig. 2 (*b*).

Temperature and Humidity

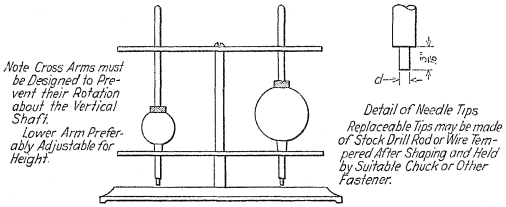
3. The temperature of the air in the vicinity of the mixing slab, the dry cement, molds, and base plates shall be maintained between 20 and 27.5 C. (68 and 81.5 F.). The temperature of the mixing water and of the moist closet or moist room shall not vary from 21 C. (70 F.) by more than plus or minus 1.7 C. (3 F.) The moist closet or moist room shall be so constructed as to provide storage facilities for test specimens at a relative humidity of not less than 90 per cent.

Preparation of Cement Paste

4. A 500-g. sample of cement shall be placed on a smooth, nonabsorbent surface and a crater formed in the center. The percentage of clean water required for normal consistency[4] shall be poured into the crater, and the cement on the outer edge shall be turned into the crater within 30 sec. by the aid of a trowel. After an additional interval of 30 sec. for the absorption of the water, during which interval the dry cement around the outside of the cone shall be lightly troweled over the remaining mixture to reduce the evaporation losses and to promote absorption, the operation shall be completed by continuous, vigorous mixing, squeezing, and kneading with the hands for 1½ min. During the operation of mixing, the hands shall be protected by snug-fitting rubber gloves.



(*a*) Pat with Top Surface Flattened for Determining Time of Setting by Gillmore Method.



(*b*) Gillmore Needles.

FIG. 2.—Gillmore Apparatus and Test Specimen.

[4] Standard Method of Test for Normal Consistency of Hydraulic Cement (A.S.T.M. Designation: C 187), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Procedure for Vicat Method

5. (*a*) *Molding Test Specimen.*—The cement paste, prepared as described in Section 4, shall be quickly formed into a ball with the gloved hands and tossed six times from one hand to the other, maintaining the hands about 6 in. apart. The ball, resting in the palm of one hand, shall be pressed into the larger end of the conical ring *G*, Fig. 1, held in the other hand, completely filling the ring with paste. The excess at the larger end shall then be removed by a single movement of the palm of the hand. The ring shall then be placed on its larger end on a glass plate *H*, and the excess paste at the smaller end sliced off at the top of the ring by a single oblique stroke of a sharp-edged trowel held at a slight angle with the top of the ring, and the top smoothed, if necessary, with a few light touches of the pointed end of the trowel. During these operations of cutting and smoothing, care shall be taken not to compress the paste. Immediately after molding, the test specimen shall be placed in the moist closet or moist room and remain there except when the determinations of time of setting are being made. The test specimens shall remain in the conical mold, supported by the plate *H*, throughout the test period.

(*b*) *Time of Setting Determination.*—The needle *D* of the rod *B*, Fig. 1, shall be lowered until it rests on top of a portion of the glass plate which projects beyond the ring *G*, and the adjustable indicator *F* set to the lower zero mark of the scale or an initial reading taken. The rod *B* shall then be raised, the needle *D* carefully brought in contact with the surface of the paste, and the rod quickly released. The *initial set* shall be said to have occurred when the needle ceases to pass a point 5 mm. above the glass plate in 30 sec. after being released; and the *final set*, when the needle does not sink visibly into the paste.

(*c*) *Precautions.*—The test specimens shall be kept in the moist closet or moist room during the test. Care shall be taken to keep the needle clean, as the collection of cement on the sides of the needle retards the penetration, while cement on the point may increase the penetration. The time of setting is affected not only by the percentage and temperature of the water used and the amount of kneading the paste received, but also by the temperature and humidity of the air, and its determination is therefore only approximate.

Procedure for Gillmore Method

6. (*a*) *Molding Test Specimen.*—From the cement paste, prepared as described in Section 4, a pat about 3 in. in diameter and $\frac{1}{2}$ in. in thickness at the center with a flat top and tapering to a thin edge, Fig. 2 (*a*) shall be made on a flat, clean glass plate about 4 in. square. In molding the pat, the cement paste shall first be flattened on the glass and the pat then formed by drawing the trowel from the outer edge toward the center, then flattening the top. After making, the pat shall be placed in the moist closet or moist room and remain there except when the determinations of time of setting are being made.

(*b*) *Time of Setting Determination.*—In determining the time of setting, the needles shall be held in a vertical position and applied lightly to the surface of the pat. The cement shall be considered to have acquired its *initial set* when the pat will bear, without appreciable indentation, the Gillmore needle $\frac{1}{12}$ in. in diameter, loaded to weigh $\frac{1}{4}$ lb. The cement shall be considered to have acquired its *final set* when the pat will bear, without appreciable indentation, the Gillmore needle $\frac{1}{24}$ in. in diameter, loaded to weigh 1 lb.

(*c*) *Precautions.*—The appropriate precautions described in Section 5 (*c*) shall be observed.

Standard Method of Test for

TENSILE STRENGTH OF HYDRAULIC-CEMENT MORTARS[1]

A.S.T.M. Designation: C 190 - 44

ADOPTED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 190; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test is intended for determining the tensile strength of hydraulic-cement mortars.

Apparatus

2. (*a*) *Scales.*—The scales used in weighing materials for the mortar mixes shall conform to the following requirements: On scales in use the permissible variation at a load of 1000 g. shall be plus or minus 1.0 g. and at a load of 1500 g. shall be plus or minus 2.0 g. The permissible variations on new scales shall be one half of these values. The sensibility reciprocal[3] shall be not greater than twice the permissible variation.

(*b*) *Weights.*—The permissible variations on weights in use in weighing materials for the mortar mixes shall be as prescribed in Table I. The permissible variations on new weights shall be one half of the values in Table I.

TABLE I.—PERMISSIBLE VARIATIONS ON WEIGHTS.

Weight, g.	Permissible Variations on Weights in Use, plus or minus, g.
1000	0.5
900	0.45
750	0.4
500	0.35
300	0.3
250	0.25
200	0.20
100	0.15
50	0.10
20	0.05
10	0.04
5	0.03
2	0.02
1	0.01

(*c*) *Sieves.*—Square-hole, woven wire cloth, No. 20 (840-micron) and No. 30 (590-micron) sieves conforming to the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11)[4] shall be used.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-1 on Cement.

[2] This method formerly constituted a portion of the Standard Methods of Sampling and Physical Testing of Portland Cement (C 77 - 40), discontinued in 1944, which latter standard was originally part of the Standard Specifications for Portland Cement (A.S.T.M. Designation: C 9) published as standard from 1904 to 1930, at which time the specifications and methods were separated and published individually.

[3] The sensibility reciprocal is a measure of the sensitiveness of a balance, and is the weight required to move the position of equilibrium of the pointer one division. For a complete definition of sensibility reciprocal, see "Specifications, Tolerances, and Regulations for Commercial Weighing and Measuring Devices," *Handbook H29*, Nat. Bureau Standards, September, 1942, pp. 87 and 88.

[4] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(*d*) *Glass Graduates.*—Glass graduates of suitable capacities (preferably large enough to measure the mixing water in a single operation) shall be made to deliver the indicated volume at 20 C. (68 F.). The permissible variation shall be plus or minus 1.0 ml. These graduates shall be subdivided to at least 1 ml. The main graduation lines shall be circles and shall be numbered. The least graduations shall extend at least one seventh of the way around, and intermediate graduations shall extend at least one fifth of the way around.

(*e*) *Briquet Molds.*—The molds for making test specimens shall be made of metal not attacked by the cement mortar, and shall have sufficient material in the sides to prevent spreading during molding. Gang molds, when used, shall be of the type shown in Fig. 1. The

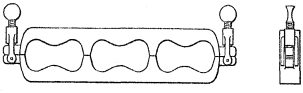


FIG. 1.—Briquet Gang Mold.

dimensions of the briquet molds shall conform to the following requirements: width of mold, between inside faces, at waist line of briquet, 1 in. with permissible variations of plus or minus 0.01 in. for old molds and plus or minus 0.005 in. for new molds; thickness of new molds measured at point of greatest thickness on either side of mold at waist line, 1 in. with permissible variations of plus 0.004 in. and minus 0.002 in.

(*f*) *Trowel.*—The trowel shall have a steel blade and be of convenient size.

(*g*) *Testing Machine.*—The testing machine shall be capable of applying the load continuously at a rate of 600 ± 25 lb. per min., with provision for adjustments of the rate of loading. The requirements for accuracy of the testing machine shall be as follows: The error for loads of not less than 100 lb. shall not exceed plus or minus 1.0 per cent for a new machine, or plus or minus 1.5 per cent for a used machine. The testing machine shall be calibrated frequently in order to determine its accuracy.

(*h*) *Clips.*—The clips for holding the tension test specimens shall be in accordance with Fig. 2.

Temperature and Humidity

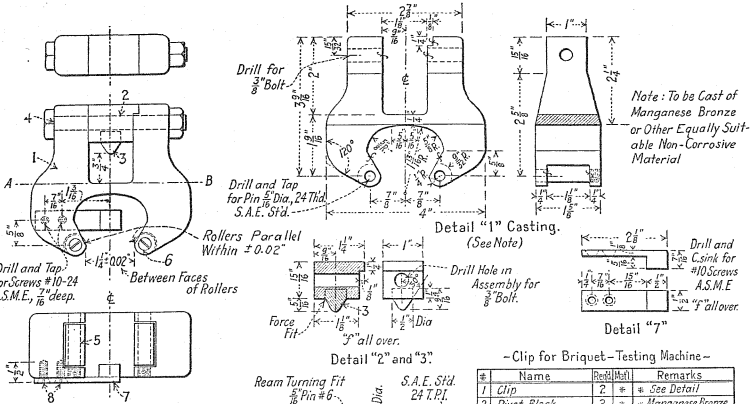
3. The temperature of the air in the vicinity of the mixing slab, the dry materials, molds, and base plates shall be maintained between 20 and 27.5 C. (68 and 81.5 F.). The temperature of the mixing water, moist closet or moist room, and water in the briquet storage tank shall not vary from 21 C. (70 F.) by more than plus or minus 1.7 C. (3 F.). The moist closet or moist room shall be so constructed as to provide storage facilities for test specimens at a relative humidity of not less than 90 per cent.

Standard Sand

4. The sand used for making test specimens shall be natural silica sand from Ottawa, Ill., graded to pass a No. 20 (840-micron) sieve and retained on a No. 30 (590-micron) sieve. This sand shall be considered standard when not more than 15 g. are retained on the No. 20 sieve, and not more than 5 g. pass the No. 30 sieve after 5 min. of continuous sieving of a 100-g. sample in the manner specified for sieving cement in the Standard Method of Test for Fineness of Hydraulic Cement by the No. 200 Sieve (A.S.T.M. Designation: C 184).[4]

Test Specimens

5. The briquet test specimens shall conform to the dimensional requirements



–Clip for Briquet–Testing Machine–

#	Name	Req'd	Matl	Remarks
1	Clip	2	*	* See Detail
2	Pivot Block	2	*	* Manganese Bronze
3	Pivot	2	Steel	Case-Hardened
4	Sq. Hd. Bolt, Hex. Nut	2	M.S.	3/8" Std. - 3 1/2" long
5	Roller	4	* *	* * Stainless Steel
6	Pin	4	* *	Ditto
7	Guide	2	* *	Ditto
8	Flat Head Mach. Scr.	4	Brass	#10-24 Thd. A.S.M.E.

FIG. 2.—Clips for Briquet Testing Machine.

shown in Fig. 3. Three or more specimens shall be made for each period of test specified.

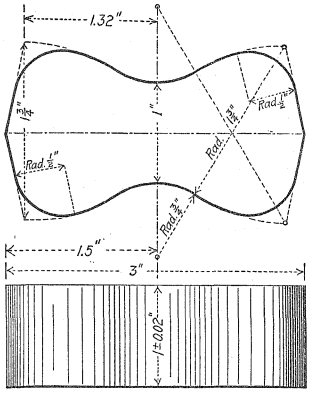


FIG. 3.—Briquet Specimen for Tensile Strength Test.

Proportioning, Consistency, and Mixing of Mortars

6. (*a*) The proportions of the standard mortar shall be 1 part cement to 3 parts standard sand by weight. The quantities of dry materials to be mixed at one time in the batch of mortar shall be not less than 1000 nor more than 1200 g. for making six briquets and not less than 1500 nor more than 1800 g. for making nine briquets. The percentage of water used in the standard mortar shall depend upon the percentage of water required to produce a neat cement paste of normal consistency from the same sample of cement and shall be as indicated in Table II, the values being in percentage of the combined dry weights of the cement and standard sand. The percentage of water required to produce a neat cement paste of normal consistency shall be determined in accordance with the Standard Method of Test for Normal Consistency of Hydraulic Cement (A.S.T.M. Designation: C 187).[4]

(*b*) The dry materials shall be weighed, placed upon a smooth nonabsorbent surface, thoroughly mixed dry, and a crater formed in the center. The proper percentage of clean water shall be poured into the crater, and the material on the outer edge turned into the crater within 30 sec. by the aid of a trowel. After an additional interval of 30 sec. for the absorption of the water, during which interval the dry mortar around the outside of the cone shall be lightly troweled over the remaining mortar to reduce the evaporation losses and to promote absorption, the operation shall be completed by continuous, vigorous mixing,

TABLE II.—PERCENTAGE OF WATER FOR STANDARD MORTARS.

Percentage of Water for Neat Cement Paste of Normal Consistency	Percentage of Water for Mortar of One Part Cement to Three Parts Standard Sand[a]
15	9.0
16	9.2
17	9.3
18	9.5
19	9.7
20	9.8
21	10.0
22	10.2
23	10.3
24	10.5
25	10.7
26	10.8
27	11.0
28	11.2
29	11.3
30	11.5

[a] When the proportions of cement to sand are other than one to three by weight, the amount of mixing water shall be calculated from the following formula, upon which Table II is based:

$$y = \tfrac{3}{4}\frac{P}{n+1} + K$$

where:

y = percentage of water required for the sand mortar,
P = percentage of water required for neat cement paste of normal consistency,
n = number of parts of sand to one of cement by weight, and
K = a constant which for the standard sand has the value 6.5.

squeezing, and kneading with the hands for 1½ min. During the operation of mixing, the hands shall be protected by snug-fitting rubber gloves.

Molding Test Specimens

7. Before being filled, the molds shall be thinly covered with a film of mineral oil. Immediately following completion of mixing the mortar, the molds, resting on unoiled glass or metal plates, shall be filled heaping full without compacting. The mortar shall then be pressed in firmly with the thumbs, applying the force 12 times to each briquet, at points to include the entire surface. The force shall be such that the simultaneous application of both thumbs will register a force of 15 to 20 lb. Each application of the thumbs shall be maintained not longer than sufficient to attain the specified force. The mortar shall then be heaped above the mold and smoothed off with a trowel. The trowel shall be drawn over the mold in such a manner as to exert a force of not more than 4 lb. The mold shall then be covered with a plane glass or metal plate oiled with mineral oil, and the mold and plates (held together with the hands) shall then be turned over, rotating the mold about its longitudinal axis. The top plate shall then be removed and the operation of heaping, thumbing, heaping, and smoothing off shall be repeated. No ramming or tamping shall be used, nor any troweling in excess of that required to smooth off the specimen.

Storage of Test Specimens

8. All test specimens, immediately after molding, shall be kept in the molds on the base plates in the moist closet or moist room for from 20 to 24 hr. with their upper surfaces exposed to the moist air but protected from dripping water. If removed from the molds before 24 hr., they shall be kept on the shelves of the moist closet or moist room until they are 24 hr. old, when the specimens, except those for the 24-hr. test, shall be immersed in clean water in storage tanks constructed of noncorroding materials. The storage water shall be kept clean by frequent changing.

Procedure

9. (*a*) The briquet specimens shall be tested immediately after their removal from the moist closet for 24-hr. specimens, and from storage water for all other specimens. If more than one specimen at a time is removed from the moist closet for the 24-hr. tests, these briquets shall be covered with a damp cloth until time of testing. If more than one specimen at a time is removed from the storage water for testing, these briquets shall be placed in a pan of water at a temperature of 21 ± 1.7 C. (70 ± 3 F.) and of sufficient depth to completely immerse each briquet until time of testing.

(*b*) Each briquet shall be wiped to a surface-dry condition, and any loose sand grains or incrustations shall be removed from the surfaces that will be in contact with the clips of the testing machine. The bearing surfaces of the clips shall be clean and free of sand, and the roller bearings shall be well oiled and maintained so as to insure freedom of turning. The stirrups supporting the clips shall be kept free of accumulations, and the pivots shall be kept in proper adjustment so that the clips may swing freely on the pivots without binding in the stirrups. The briquets shall be carefully centered in the clips and the load applied continuously at the rate of 600 ± 25 lb. per min.

Faulty Briquets and Retests

10. Briquets that upon removal from the molds do not conform to the requirements for thickness and width at the waist line (Fig. 3), or that are mani-

festly faulty, or that give strengths differing by more than 15 per cent from the average value of all test specimens made from the same sample and tested at the same period, shall not be considered in determining the tensile strength (Note). After discarding briquets or strength values, if less than two strength values are left for determining the tensile strength at any given period, a retest shall be made.

NOTE.—Reliable strength results depend upon careful observance of all of the specified requirements and procedures. Erratic results at a given test period indicate that some of the requirements and procedures have not been carefully observed; for example, those covering the testing of the briquets, as prescribed in Section 9 (*b*).

Standard Method of Test for

COMPRESSIVE STRENGTH OF HYDRAULIC-CEMENT MORTARS[1]

A.S.T.M. Designation: C 109 - 44

ADOPTED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 109; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test is intended for determining the compressive strength of hydraulic-cement mortars.

Apparatus

2. (*a*) *Scales.*—The scales used in weighing materials for mortar mixes shall conform to the following requirements: On scales in use the permissible variation at a load of 2000 g. shall be plus or minus 2.0 g. The permissible variation on new scales shall be one half of this value. The sensibility reciprocal[3] shall be not greater than twice the permissible variation.

(*b*) *Weights.*—The permissible variations on weights in use in weighing materials for mortar mixes shall be as prescribed in Table I. The permissible variations on new weights shall be one half of the values in Table I.

TABLE I.—PERMISSIBLE VARIATIONS ON WEIGHTS.

Weight, g.	Permissible Variations on Weights in Use, plus or minus, g.
1000	0.5
900	0.45
750	0.4
500	0.35
300	0.3
250	0.25
200	0.20
100	0.15
50	0.10
20	0.05
10	0.04
5	0.03
2	0.02
1	0.01

(*c*) *Sieves.*—Square-hole, woven wire cloth No. 100 (149-micron), No. 50 (297-micron), No. 30 (590-micron) and No. 16 (1190-micron) sieves conforming to the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11)[4] shall be used.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-1 on Cement.

[2] Prior to adoption as standard, this method was published as tentative from 1934 to 1944, being revised in 1937, 1943, and 1944.

[3] The sensibility reciprocal is a measure of the sensitiveness of a balance, and is the weight required to move the position of equilibrium of the pointer one division. For a complete definition of sensibility reciprocal, see "Specifications, Tolerances, and Regulations for Commercial Weighing and Measuring Devices," *Handbook H29*, Nat. Bureau Standards, September, 1942, pp. 87 and 88.

[4] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(*d*) *Glass Graduates.*—Glass graduates of suitable capacities (preferably large enough to measure the mixing water in a single operation) shall be made to deliver the indicated volume at 20 C. (68 F.). The permissible variation shall be plus or minus 2 ml. These graduates shall be subdivided to at least 5 ml. The main graduation lines shall be circles and shall be numbered. The least graduations shall extend at least one seventh of the way around, and intermediate graduations shall extend at least one fifth of the way around.

(*e*) *Specimen Molds.*—Molds for the 2-in. cube test specimens shall be tight fitting. The parts of the molds when assembled shall be positively held together. The molds shall be made of hard metal not attacked by the cement mortar. For new molds the Rockwell hardness number of the metal shall be not less than B 55. There shall be sufficient material in the sides of the molds to prevent spreading or warping. The interior faces of the molds shall be plane surfaces with a permissible variation of 0.001 in. for new molds and 0.002 in. for molds in use. The distances between opposite faces shall be 2 ± 0.005 in. for new molds, and 2 ± 0.02 in. for molds in use. The height of the molds, measured separately for each cube compartment, shall be 2 in. with permissible variations of plus 0.01 in. and minus 0.005 in. for new molds, and plus 0.01 in. and minus 0.015 in. for molds in use. The angle between adjacent interior faces, and between interior faces and top and bottom planes of the mold shall be 90 ± 0.5 deg. measured at points slightly removed from the intersection of the faces.

(*f*) *Mixing Bowl.*—The mixing bowl shall be of enamelware or other nonabsorptive, noncorroding material and shall have a capacity of 6 to 8 qt.

(*g*) *Flow Table.*—The flow table apparatus shall consist of a rigid metal frame and a circular rigid table 10 in. in diameter with a shaft attached perpendicular to the table top. The table with attached shaft shall be mounted on the frame in such a manner that it can be raised and dropped vertically through a fixed height of ½ in. by means of a rotated cam. The table top shall have a plane surface and be of noncorrodible metal. The table and the attached shaft shall weigh 9 ± 0.1 lb. The end of the shaft shall not strike upon the cam at the end of the drop. The surfaces of the table and the frame which come into contact at the end of the drop shall be plane and parallel with the upper surface of the table and the material of these parts shall be hard metal to prevent cushioning effect. The contact faces of the cam and the shaft shall be such that the table does not rotate more than one revolution during the 25 drops. The frame shall be attached rigidly to a concrete pedestal which shall be attached rigidly to the floor. The concrete pedestal shall be at least 8 in. square or in diameter at the top and at least 25 in. in height with a base suitable for rigid attachment to the floor and shall weigh at least 100 lb. The table top, after the frame has been mounted on the pedestal, shall be level along any two diameters at right angles to each other in both the raised and the lowered position.

(*h*) *Flow Mold.*—The flow mold shall be made of noncorrodible material and shall be 4 in. in inside diameter at the base, 2.75 in. in inside diameter at the top, and 2 in. in height.

(*i*) *Tamper.*—The tamper shall be made of a nonabsorptive, nonabrasive material such as medium-hard rubber or seasoned oak wood rendered nonabsorptive by immersion for 15 min. in paraffin at approximately 200 C. (392 F.), and shall have a cross-section of ½ by 1 in. and a convenient length (5 or 6 in.).

(*j*) *Trowel.*—The trowel shall have a steel blade 4 to 6 in. in length, with straight edges.

(*k*) *Testing Machine.*—The testing machine may be either the hydraulic or the screw-type with sufficient opening between the upper bearing surface and the lower bearing surface of the machine to permit the use of verifying apparatus. The load applied to the test specimen shall be indicated with an accuracy of plus or minus 1.0 per cent. The upper bearing shall be a spherically seated, hardened metal block firmly attached at the center of the upper head of the machine. The center of the sphere shall lie at the center of the surface of the block in contact with the specimen. The block shall be closely held in its spherical seat, but shall be free to turn in any direction. The diagonal or diameter[5] of the bearing surface shall be only slightly greater than the diagonal of the face of the 2-in. cube in order to facilitate accurate centering of the specimen. A hardened metal bearing block shall be used beneath the specimen to minimize wear of the lower platen of the machine. The bearing block surfaces intended for contact with the specimen shall have a Rockwell hardness number not less than C 60. These surfaces shall not depart from plane surfaces by more than 0.0005 in. when the blocks are new and shall be maintained within a permissible variation of 0.001 in.

Temperature and Humidity

3. The temperature of the air in the vicinity of the mixing slab, the dry materials, molds, base plates, and mixing bowl, shall be maintained between 20 and 27.5 C. (68 and 81.5 F.). The temperature of the mixing water, moist closet or moist room, and water in the storage tank shall not vary from 21 C. (70 F.) by more than plus or minus 1.7 C. (3 F.). The moist closet or moist room shall be so constructed as to provide storage facilities for test specimens at a relative humidity of not less than 90 per cent.

Graded Standard Sand

4. The sand (Note 1) used for making test specimens shall be natural silica sand from Ottawa, Ill., graded as follows:

Sieve	Percentage Retained
No. 100 (149-micron)	98 ± 2
No. 50 (297-micron)	72 ± 5
No. 30 (590-micron)	2 ± 2
No. 16 (1190-micron)	none

Sieve Analysis of Sand

5. (*a*) For checking the grading of the sand, a sieving test of the sand shall be made on each of the four sieves specified in Section 2 (*c*). Samples of sand for the sieve tests shall be quartered from a sample of about 700 g. obtained by the method of quartering the contents of a full sack (100 lb.) which have been thoroughly mixed and the pile flattened or spread out to minimize segregation during quartering.

(*b*) The test on each sieve shall be made with 100 g. of sand. The sieve shall be thoroughly clean and dry. The sieving operation shall be performed in the manner specified for sieving cement in the Standard Method of Test for Fineness of Hydraulic Cement by the No. 200 Sieve (A.S.T.M. Designation: C 184),[4] except that sieving shall be continued until not more than 0.5 g. passes through in 1 min. of continuous sieving. The weight of the residue on the sieve shall be expressed as a percentage of the weight of the original sample.

[5] A diameter of 3⅛ in., which is large enough for testing 3 by 6-in. cylinders, is satisfactory provided that the lower bearing block has a diameter slightly greater than the diagonal of the face of the 2-in. cube but not more than 2.9 in., and is centered with respect to the upper bearing block and held in position by suitable means.

Number of Specimens

6. Three or more specimens shall be made for each period of test specified.

Preparing Specimen Molds

7. The interior faces of the specimen molds shall be thinly covered with mineral oil or light cup grease. The contact surfaces of the halves of each mold shall be thinly covered with a heavy mineral oil or light cup grease such as petrolatum. After assembling the molds, excess oil or grease shall be removed from the interior faces and the top and bottom surfaces of each mold. Molds shall then be set on plane, nonabsorptive base plates which have been thinly coated with mineral oil, petrolatum, or light cup grease. A mixture of 3 parts of paraffin to 5 parts of rosin by weight, heated between 110 and 120 C. (230 and 248 F.), shall be applied at the outside contact lines of the molds and base plates so that watertight joints are effected between the molds and the base plates (Note 2).

Proportioning, Consistency, and Mixing of Mortars

8. (*a*) The proportions of dry materials of the standard mortar shall be one part of cement to 2.75 parts of graded standard sand by weight. The quantities of dry materials to be mixed at one time in the batch of mortar for making six cubes shall be 500 g. of cement and 1375 g. of graded standard sand. The quantities of dry materials to be mixed at one time in the batch of mortar for nine cubes shall be 700 g. of cement and 1925 g. of graded standard sand. The amount of mixing water, measured in milliliters, shall be such as to produce a flow of between 100 and 115 as determined in accordance with Section 9 and shall be expressed as a percentage by weight of the cement.

(*b*) Mixing shall be done in a bowl (Section 2 (*f*)) by vigorous and continuous stirring, squeezing, and kneading with one hand which shall be protected by a snug-fitting rubber glove. The materials for a batch shall be introduced in the following manner:

(*1*) Place the water in the bowl, which has just been wiped with a damp cloth or damp sponge,

(*2*) Add the cement to the water and mix for 30 sec.,

(*3*) Add approximately one half of the testing sand and mix for 30 sec., and

(*4*) Add the remainder of the sand and mix for 1½ min.

Determination of Flow

9. The flow-table top shall be carefully wiped dry and the flow mold placed at the center. Immediately after completing the mixing operation, the mold shall be filled with mortar gently pressed into place by the finger tips to insure uniform filling. The mortar shall then be cut off to a plane surface, flush with the top of the mold, by drawing the straight edge of a trowel (held nearly perpendicular to the mold) with a sawing motion across the top of the mold. The mold shall be lifted away from the mortar 30 sec. after completing the mixing operation. Immediately the table shall be dropped through a height of ½ in., 25 times in 15 sec. The flow is the resulting increase in diameter of the mortar mass, expressed as a percentage of the original diameter. Trial mortars shall be made with varying percentages of water[6] until the specified flow is obtained. Each trial shall be made with fresh mortar. The batch of mortar upon which flow has been determined shall not be used for molding specimens.

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[6] As a guide for the initial trial mortar, the percentage of water to produce the specified flow will be about 53 per cent for portland cement containing air-entraining material and about 55 per cent for portland cement not containing air-entraining material.

Molding Test Specimens

10. Immediately following completion of the mixing, the mortar adhering to the mixing glove shall be removed by striking the hand against the side or edge of the mixing bowl. The glove shall then be removed. A layer of mortar about 1 in. in thickness shall be placed in all of the cube compartments. Then the mortar in each cube compartment shall be tamped (Section 2 (*i*)) 32 times in about 10 sec. in four rounds, each round to be at right angles to the other and consisting of eight adjoining strokes over the surface of the specimen, as illustrated in Fig. 1. The tamping pressure shall be just sufficient to insure uniform filling of the molds. The four rounds of tamping (32 strokes) of the mortar shall be completed in one cube before going to the next. When the tamping of the first layer in all of the cube compartments is completed, the compartments shall be filled with the remaining mortar and then tamped as specified for the first layer. On completion of the tamping, the tops of all cubes should extend slightly above the tops of the molds. The mortar which has been forced out onto the tops of the molds shall be brought in with a trowel and the cubes smoothed off by drawing the flat side of the trowel (with the leading edge slightly raised) once across the top of each cube at right angles to the length of the mold. The mortar shall then be cut off to a plane surface flush with the top of the mold by drawing the straight edge of the trowel (held nearly perpendicular to the mold) with a sawing motion over the length of the mold.

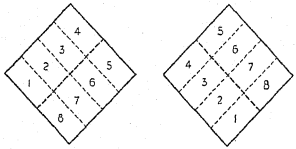


FIG. 1.—Order of Tamping in Molding of Test Specimens.

Storage of Test Specimens

11. All test specimens, immediately after molding, shall be kept in the molds on the base plates in the moist closet or moist room from 20 to 24 hr. with their upper surfaces exposed to the moist air but protected from dripping water. If removed from the molds before 24 hr., they shall be kept on the shelves of the moist closet or moist room until they are 24 hr. old, when the specimens, except those for the 24-hr. test, shall be immersed in clean water in storage tanks constructed of noncorroding materials. The storage water shall be kept clean by frequent changing.

Procedure

12. (*a*) Testing of the cube specimens shall be carried out immediately after their removal from the moist closet for 24-hr. specimens, and from storage water for all other specimens. If more than one specimen at a time is removed from the moist closet for the 24-hr. tests, these cubes shall be covered with a damp cloth until time of testing. If more than one specimen at a time is removed from the storage water for testing, these cubes shall be placed in a pan of water at a temperature of 21 ± 1.7 C. (70 ± 3 F.) and of sufficient depth to completely immerse each cube until time of testing.

(*b*) Each cube shall be wiped to a surface-dry condition, and any loose sand grains or incrustations shall be removed from the faces that will be in contact with the bearing blocks of the testing

machine. These faces shall be checked by application of a straightedge (Note 3). If appreciable curvature is present, the face or faces shall be ground to plane surfaces or the specimen shall be discarded.

(*c*) The load shall be applied to cube faces that were in contact with the true plane surfaces of the mold. The cube shall be carefully placed in the testing machine below the center of the upper bearing block. No cushioning or bedding materials shall be used. The loading up to 50 per cent (25 per cent for expected maximum loads of less than 4000 lb. at ages of 7 days or less) of the expected maximum load may be applied at any convenient rate, after which the specimen shall be loaded continuously to failure at a rate or rates which shall at no time be less than 1000 nor more than 6000 psi. per min.[7]

Calculation

13. The total maximum load indicated by the testing machine shall be recorded, and the compressive strength calculated in pounds per square inch. If the cross-sectional area of a cube varies more than 0.06 sq. in. from 4.00 sq. in., the actual area shall be used for the calculation of the compressive strength.

Faulty Cubes and Retests

14. Cubes that are manifestly faulty, or that give strengths differing by more than 10 per cent from the average value of all test specimens made from the same sample and tested at the same period, shall not be considered in determining the compressive strength (Note 4). After discarding cubes or strength values, if less than two strength values are left for determining the compressive strength at any given period, a retest shall be made.

EXPLANATORY NOTES

NOTE 1: *Segregation of Graded Sand.*—The graded standard sand should be handled in such a manner as to prevent segregation, since variations in the grading of the sand cause variations in the consistency of the mortar. In emptying sacks of sand into bins or in scooping sand out of bins or sacks, care should be exercised to prevent the formation of mounds of sand or craters in the sand, down the slopes of which the coarser particles will roll. Bins should be of sufficient size to permit these precautions. Devices for drawing the sand from bins by gravity should not be used.

NOTE 2: *Watertight Molds.*—The mixture of paraffin and rosin specified for sealing the joints between molds and base plates may be found difficult to remove when molds are being cleaned. Use of straight paraffin is permissible if a watertight joint is secured, but due to the low strength of paraffin it should be used only when the mold is not held to the base plate by the paraffin alone. A watertight joint may be secured with paraffin alone by slightly warming the mold and base plate before brushing the joint. Molds so treated should be allowed to return to the specified temperature before use.

NOTE 3: *Cube Faces.*—Results much lower than the true strength will be obtained by loading faces of the cube which are not truly plane surfaces. Therefore, it is essential that cube molds be kept scrupulously clean, as otherwise large irregularities in the surfaces will occur. Instruments for cleaning of molds should always be softer than the metal in the molds to prevent wear. In case grinding of cube faces is necessary, it can be accomplished best by rubbing the cube on a sheet of fine emery paper or cloth glued to a plane surface,

[7] While the data presented in the report of the 1933 cooperative tests (see the Report of Working Committee on Plastic Mortar Tests for Portland Cement, of Committee C-1 on Cement, *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, p. 322 (1934)) do not give conclusive evidence on effect of rate of loading, the Working Committee favors the use of a uniform rate of loading of about 4000 psi. per min. (1000 psi. per min. for low-heat portland cements at ages of 7 days or less).

using only a moderate pressure. Such grinding is tedious for more than a few thousandths of an inch; where more than this is found necessary, it is recommended that the cube be discarded.

NOTE 4.—Reliable strength results depend upon careful observance of all of the specified requirements and procedures. Erratic results at a given test period indicate that some of the requirements and procedures have not been carefully observed; for example, those covering the testing of the cubes as prescribed in Section 12 (*b*) and (*c*). Improper centering of cubes resulting in oblique fractures or lateral movement of one of the heads of the testing machine during loading will often cause lower strength results. A cube so broken shall be considered as "manifestly faulty" if its strength differs by more than 10 per cent from the average of all test specimens made from the same sample and tested at the same period.

Standard Specifications for
QUICKLIME FOR STRUCTURAL PURPOSES[1]

A.S.T.M. Designation: C 5 - 26

ADOPTED, 1926.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 5; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover all classes of quicklime, such as crushed lime, granular lime, ground lime, lump lime, pebble lime, and pulverized lime, used for structural purposes.

Chemical Composition

2. The quicklime shall conform to the following requirements as to chemical composition, calculated to the nonvolatile basis:

	Calcium Lime	Magnesium Lime
Calcium oxide, min., per cent	75	..
Magnesium oxide, min., per cent	..	20
Calcium and magnesium oxides, min., per cent	95	95
Silica, alumina, and oxide of iron, max., per cent	5	5
Carbon dioxide, max., per cent:		
If sample is taken at the place of manufacture	3	3
If sample is taken at any other place	10	10

Residue

3. The quicklime shall contain not more than 15 per cent by weight of residue.

Sampling, Inspection, etc.

4. The sampling, inspection, rejection, retesting, packing, and marking shall be conducted in accordance with the Standard Methods of Sampling, Inspection, Packing, and Marking of Quicklime and Lime Products (A.S.T.M. Designation: C 50) of the American Society for Testing Materials.[3]

Methods of Testing

5. The properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(*a*) *Chemical Analysis.*—Standard Methods of Chemical Analysis of Limestone, Quicklime, and Hydrated Lime (A.S.T.M. Designation: C 25).[3]

(*b*) *Physical Tests.*—Tentative Methods of Physical Testing of Quicklime and Hydrated Lime (A.S.T.M. Designation: C 110).[3]

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-7 on Lime.

[2] Prior to their present adoption as standard, these specifications were published as tentative from 1913 to 1915, being revised in 1914 and 1915. They were adopted in 1915 but withdrawn and republished as tentative from 1921 to 1926, being revised in 1922 and 1924. Editorially revised and rearranged in 1939.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

APPENDIX

Quicklime can never be used as such for structural purposes; it must always be slaked first. Since the method of slaking is an important factor in determining the quality of the finished product, the following directions for the preparation of lime putty are given, not as a part of the specifications, but as information for the further protection of the purchaser.

A1. *Introduction.*—Different kinds of lime vary considerably in the way in which they behave with water. A little supervision over the operation of slaking will amply pay for itself by insuring the production of the greatest possible quantity and the best possible quality of putty. To find out how to slake a new lot of lime, it is safest to try a little of it and see how it works. Since different lots of the same brand of lime vary somewhat, and since the weather conditions at the time have a decided influence, it is wise to try a sample from each lot used, whether familiar with the brand or not.

A2. *Classification of Limes.*—In a bucket, put two or three lumps of lime about the size of one's fist, or, in the case of granular lime, an equivalent amount. Add sufficient water to just barely cover the lime, and note how long it takes for slaking to begin. Slaking has begun when pieces split off from the lumps or when the lumps crumble. Water of the same temperature should be used for test and field practice.

If slaking begins in less than 5 min., the lime is quick slaking; from 5 to 30 min., medium slaking; over 30 min., slow slaking.

A3. *Directions for Slaking.*—For quick-slaking lime, always add the lime to the water, not the water to the lime. Have sufficient water at first to cover all the lime completely. Have a plentiful supply of water available for immediate use—a hose throwing a good stream, if possible. Watch the lime constantly. At the slightest appearance of escaping steam, hoe thoroughly and quickly, and add enough water to stop the steaming. Do not be afraid of using too much water with this kind of lime.

For medium-slaking lime, add the water to the lime. Add enough water so that the lime is about half submerged. Hoe occasionally if steam starts to escape. Add a little water now and then if necessary to prevent the putty from becoming dry and crumbly. Be careful not to add any more water than required, and not too much at a time.

For slow-slaking lime, add enough water to the lime to moisten it thoroughly. Let it stand until the reaction has started. Cautiously add more water, a little at a time, taking care that the mass is not cooled by the fresh water. Do not hoe until the slaking is practically complete. If the weather is very cold, it is preferable to use hot water, but if this is not available, the mortar box may be covered in some way to retain the heat.

A4. *Preparation of Putty for Use.*—After slaking, the putty shall be prepared for use, as follows:

(*a*) *White Coat.*—After the action has ceased, run off the putty through a No. 10 (2000-micron) sieve and store for a minimum of two weeks.

(*b*) *Base Coats.*—After the action has ceased, run off the putty through a No. 8 (2380-micron) sieve. Add sand up to equal parts by weight, all of the hair required, and store for a minimum of two weeks.

(*c*) *Masons' Mortar.*—After the action has ceased, add part or all of the sand required, and store for a minimum of 24 hr.

Standard Specifications for

NORMAL FINISHING HYDRATED LIME[1]

A.S.T.M. Designation: C 6 – 44

ADOPTED, 1924; REVISED, 1931, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 6; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover one type of finishing hydrated lime which is suitable for use in the scratch, brown, and finish coats of plaster, for stucco, mortar, and as an addition to portland-cement concrete. Lime sold under these specifications shall be designated type N—normal finishing hydrate.

Chemical Composition

2. Type N—normal finishing hydrate shall conform to the following requirements as to chemical composition, calculated to the nonvolatile basis:

Calcium and magnesium oxides, min., per cent	95
Carbon dioxide, max., per cent:	
If sample is taken at the place of manufacture	5
If sample is taken at any other place	7

Residue

3. The percentage residue of type N—normal finishing hydrate shall conform to the following requirements:

Residue retained on No. 30 (590-micron) sieve, max., per cent	0.5
Residue retained on No. 200 (74-micron) sieve, max., per cent	15

Soundness

4. Type N—normal finishing hydrate shall pass the test for soundness. If the steam has no visible effect on the pat, the sample shall be reported as being "sound." If the pat disintegrates, the sample shall be reported as being unsound and the shipment rejected. If the pat cracks, pops, or shows other minor defects, it shall not be reported as either sound or unsound, but its behavior shall be noted.

Plasticity

5. The putty made from type N—normal finishing hydrate shall have a plasticity figure of not less than 200 when soaked for a period of not less than 16 hr. nor more than 24 hr.

Sampling, Inspection, etc.

6. The sampling, inspection, rejection, retesting, packing, and marking

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-7 on Lime.

[2] Prior to their present adoption as standard, these specifications were published as tentative from 1913 to 1915, being revised in 1914 and 1915. They were adopted in 1915, revised in 1919, but withdrawn and republished as tentative from 1921 to 1924, being revised in 1922, 1923, and 1924.

shall be conducted in accordance with the Standard Methods of Sampling, Inspection, Packing, and Marking of Quicklime and Lime Products (A.S.T.M. Designation: C 50).[2]

Methods of Testing

7. The properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(*a*) *Chemical Analysis.*—Standard Methods of Chemical Analysis of Limestone, Quicklime, and Hydrated Lime (A.S.T.M. Designation: C 25).[2]

(*b*) *Physical Tests.*—Tentative Methods of Physical Testing of Quicklime and Hydrated Lime (A.S.T.M. Designation: C 110).[2]

[2] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for

HYDRAULIC HYDRATED LIME FOR STRUCTURAL PURPOSES[1]

A.S.T.M. Designation: C 141 – 42

ADOPTED, 1942.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 141; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. (*a*) These specifications cover hydraulic hydrated lime for structural purposes.

(*b*) Hydraulic hydrated lime may be used for scratch or brown coat of plaster, for stucco, for mortar, and as sole cementitious material in concrete, or in portland-cement concrete either as blend, amendment, or admixture.

Definition

2. *Hydraulic Hydrated Lime.*—An hydraulic hydrated lime is the hydrated dry cementitious product obtained by calcining a limestone containing silica and alumina to a temperature short of incipient fusion so as to form sufficient free lime (CaO) to permit hydration and at the same time leaving unhydrated sufficient calcium silicates to give the dry powder, meeting the requirements herein prescribed, its hydraulic properties.

(*a*) High Calcium Hydraulic Hydrated Lime is a lime that contains not more than 5 per cent magnesium oxide (of the nonvolatile portion).

(*b*) Magnesium Hydraulic Hydrated Lime is a lime containing more than 5 per cent magnesium oxide (of the nonvolatile portion).

Chemical Composition

3. The hydraulic hydrated lime shall conform to the following requirements as to chemical composition, calculated to the nonvolatile basis:

	Minimum	Maximum
Calcium and magnesium oxides (CaO and MgO), per cent	60	70
Silica (SiO_2), per cent	16	26
Iron and aluminum oxides (Fe_2O_3 and Al_2O_3), per cent		12
Carbon dioxide (CO_2), per cent		5

Fineness

4. The sample shall leave a residue of not more than 0.5 per cent on a No. 30 (590-micron) sieve and not more than 10 per cent on a No. 200 (74-micron) sieve when tested as described in Section 18.

Time of Setting

5. The neat hydraulic hydrated lime

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-7 on Lime.

[2] Prior to adoption as standard, these specifications were published as tentative from 1938 to 1942. Editorially revised in 1944.

paste mixed to normal consistency shall not develop an initial set in less than 2 hr. as determined by the Gillmore needle method. Final set shall be attained within 48 hr.

Soundness

6. Pats of neat hydraulic hydrated lime made and stored, as described in Section 21 shall be firm and hard and show no signs of distortion, cracking, or disintegration under both conditions of storage. Mortar strength specimens at the time of test shall show no signs of distortion, cracking, or disintegration.

Compressive Strength

7. The average compressive strength of not less than three 2-in cubes, made, stored, and tested in accordance with Sections 22 to 28, shall be not less than 175 psi. at the age of 7 days. The average strength attained at 28 days shall be not less than that attained at 7 days.

Packaging and Marking

8. The hydraulic hydrated lime shall be delivered in suitable containers with the brand and name of the manufacturer and the net weight plainly marked thereon. A bag shall contain 62.5 lb. net. A barrel shall contain 250 lb. net. All packages shall be in good condition at the time of inspection. Packages varying more than 10 per cent from the weight marked thereon may be rejected, and if the average weight of packages in any shipment, as shown by weighing 50 packages taken at random, is less than the weight marked thereon, the entire shipment may be rejected.

Storage

9. The hydraulic hydrated lime shall be stored in such a manner as to permit easy access for proper inspection and identification of each shipment, and in a suitable weather-tight building that will protect the hydraulic hydrated lime from damage.

Inspection

10. Every facility shall be provided the purchaser for sampling and inspection either at the place of manufacture or at the destination, as may be specified by the purchaser.

Rejection

11. The hydraulic hydrated lime may be rejected if it fails to meet any of the requirements of these specifications. However, hydraulic hydrated lime failing to meet the 7-day requirements may be held awaiting the result of the 28-day tests before rejection. At least ten days from the time of sampling shall be allowed for completion of the 7-day test, and at least thirty-one days shall be allowed for completion of the 28-day test.

SAMPLING AND METHODS OF TESTING

Size and Number of Samples

12. Each sample selected for purpose of tests shall weigh at least 5 lb. and shall represent not more than 200 bbl. If only one sample is taken it shall weigh at least 10 lb.

Shipment of Samples

13. Samples shall be shipped and stored in airtight, moistureproof containers.

Chemical Analysis

14. Analyses for chemical composition shall be carried out in accordance with the Standard Methods of Chemical Analysis of Limestone, Quicklime, and Hydrated Lime (A.S.T.M. Designation: C 25) of the American Society for Testing Materials.[3]

Standard Temperature

15. The standard temperature shall

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

be 70 F. (21 C.). The air of the laboratory, the materials, the mixing water, the moist closet, and storage tanks shall be maintained as nearly as practicable at the standard temperature and shall not vary therefrom more than 5 F. (3 C.).

Preliminary Treatment of Samples

16. The sample shall be given a preliminary sieving by being passed through a No. 20 sieve in order thoroughly to mix the sample, break up lumps, and remove foreign materials.

Fineness Test

17. (*a*) *Apparatus.*—The No. 30 (590-micron) and No. 200 (74-micron) sieves shall conform to the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials.[3]

(*b*) *Procedure.*—A 100-g. sample of the material as received shall be placed on a No. 30 sieve, which shall be nested above a No. 200 sieve. The material shall be washed by means of a stream of water from a faucet (Note 1). The washing shall be continued until the water coming through the sieve is clear, but in no case shall the washing be continued for more than 15 min. (Note 2). The residue on both sieves shall be dried to constant weight at a temperature between 212 and 248 F. (100 and 120 C.). The weights of material retained shall be calculated as a percentage of the original sample, the weight of the material retained on the No. 30 sieve being added to the weight of the material retained on the No. 200 sieve to obtain the correct weight of the material retained on the No. 200 sieve.

NOTE 1.—A piece of rubber tubing attached to a water faucet may be used for the washing. The velocity of the water, which may be increased by pinching the tubing, shall not be sufficient to cause any splashing of the sample over the sides of the sieve.

NOTE 2.—Care shall be taken not to let water accumulate on the No. 200 sieve, because the openings will become clogged and the operation cannot be completed in 15 min.

Mixing Paste[4]

18. Hydraulic hydrated lime pastes shall be mixed in accordance with Section 2 (*a*) to (*c*) and Section 4 (*a*) of the Standard Method of Test for Normal Consistency of Hydraulic Cement (A.S.T.M. Designation: C 187),[3] except that the operation of mixing, squeezing, and kneading with the hands shall be continued for 5 min.

Normal Consistency[4]

19. Normal consistency shall be determined by the Vicat apparatus in accordance with Standard Method C 187,[3] except that the mixing of the paste shall be performed in accordance with Section 18 of these specifications.

Time of Setting Test[4]

20. Time of setting shall be determined by the Gillmore needle method in accordance with the Standard Methods of Test for Time of Setting of Hydraulic Cement by the Vicat or Gillmore Needles (A.S.T.M. Designation: C 191),[3] except that the mixing of the paste shall be performed in accordance with Section 18 of these specifications. The hydraulic hydrated lime pats shall be stored during the test in a damp closet maintained at a relative humidity of 90 per cent or more.

Soundness Test

21. Two pats of hydraulic hydrated lime paste of normal consistency (Section 19) shall be used for making soundness tests. After the first 48 hr. in the damp closet one of the pats shall be stored in the air of the laboratory for 5 days and the other shall be immersed for 5 days in clean running water in storage tanks of suitable materials. Unsoundness is manifested by change in volume, which causes distortion, cracking, checking, or disintegration. Pats

[4] This section was editorially revised in 1944.

improperly made or exposed to drying may develop what is known as shrinkage cracks within the first 24 hr. The shrinkage cracks are not an indication of unsoundness. The mere failure of the pats to remain on the glass or the cracking of the glass to which the pats are attached do not indicate unsoundness. Should the pat leave the plate, distortion may be detected with a straightedge applied to the surface that was in contact with the plate, provided the glass plate was flat before test.

Compressive Strength Test

22. *Standard Sand.*[5]—The sand used for making test specimens shall be natural silica sand from Ottawa, Ill., graded to pass a No. 20 (840-micron) sieve and retained on a No. 30 (590-micron) sieve.[6] This sand shall be considered standard when not more than 15 g. are retained on the No. 20 sieve, and not more than 5 g. pass the No. 30 sieve after 5 min. of continuous sieving of a 100-g. sample in the manner specified for sieving cement in the Standard Method of Test for Fineness of Hydraulic Cement by the No. 200 Sieve (A.S.T.M. Designation: C 184).[3]

23. *Test Specimen.*—The compression test specimen shall be a 2-in. cube. The molds shall be of noncorrodible metal and shall be so designed as to prevent spreading during molding. Molds shall be oiled with a mineral oil before using.

24. (*a*) *Proportions for Standard Mortar.*[4]—The mortar shall consist of 1 part of hydraulic hydrated lime to 3 parts of standard Ottawa sand, by weight.

(*b*) *Preparation of Standard Mortar.*—The mortar shall be mixed in a nonabsorbent bowl of about 1-gal. capacity. A measured quantity of water shall be poured into the bowl which previously has been wiped with a damp cloth. A 500-g. sample of the hydraulic hydrated lime shall then be added and stirred into the water with the fingers of one hand until all of the hydraulic hydrated lime is wetted. Approximately 800 g. of sand shall then be added and the stirring continued for 30 sec. The remainder of 1500 g. of sand shall then be added and the mortar mixed for 75 sec. by vigorous and continued stirring, squeezing, and kneading with one hand. The mortar shall be allowed to stand for 60 sec. and then mixed for another 60 sec. During the operation of mixing, the hands shall be protected by rubber gloves. The top of the flow table (see Paragraph (*c*)) shall be carefully wiped dry and the flow mold placed at the center and filled with the mortar. In filling the mold the mortar shall not be rammed, but gently puddled to insure uniform filling. The mortar shall be smoothed off level with the top of the mold by aid of a trowel and the mold then removed. Immediately, the table shall be dropped through a height of ½ in., 25 times in 15 sec. The flow is the resulting increase in diameter of the mortar mass, expressed as a percentage of the original diameter. The mortar shall be of standard consistency when the flow is 100 to 115. Trial mortars shall be made with varying percentages of water until the standard consistency is obtained. Each trial shall be made with fresh mortar. The quantity of water shall be expressed as a percentage of the weight of the combined dry materials.

(*c*) *Flow Table.*—The flow table apparatus shall consist of a rigid frame with a flat circular top, so mounted on a vertical shaft that it can be raised and dropped ½ in. by means of a cam. The top shall be of noncorrodible metal,

[5] This section was added editorially in 1944.

[6] Detailed requirements for these sieves are given in the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

10 in. in diameter, and with the attached shaft shall weigh 9 lb. The mold shall be of noncorrodible material, 4 in. in inside diameter at the base, $2\frac{3}{4}$ in. in inside diameter at the top, and 2 in. in height.

25. *Molding of Test Specimens.*—The surfaces of the molds and plates to be in contact with the cube specimens shall be oiled with a medium viscosity oil. The molds shall be half filled with mortar, the mortar puddled into place with the finger tips of the gloved hand, the mold then filled to overflowing, and the mortar again puddled with the finger tips. The mortar shall then be troweled off flush with the top of the molds.

26. *Storage of Test Specimens.*—All test specimens, immediately after molding, shall be kept in the molds on plane plates in a damp closet, maintained at a relative humidity of 90 per cent or more, for from 48 to 52 hr. in such a manner that the upper surfaces shall be exposed to the moist air. The specimens shall then be removed from the molds and placed in the air of the laboratory for 5 days in such a manner as to allow free circulation of air around at least five faces of the specimens. At the age of 7 days the specimens for the 28-day tests shall be immersed in clean running water in storage tanks of noncorrodible materials.

27. *Testing of Specimens.*—The compressive strength test may be made with any type of verified compression testing machine of suitable capacity. Those faces of the cube specimen which when molded were in contact with the sides of the mold shall be placed in contact with the surfaces through which the load is applied. A spherical-seated bearing block shall be used between the upper face of the specimen and the head of the testing machine. The center of the sphere shall lie in that surface of the bearing block which is in contact with the specimen, and the radius of the sphere shall be $1\frac{1}{2}$ in. The specimen shall be carefully centered in the testing machine and below the center of the bearing block. Cushioning materials shall not be used. The load shall be applied uniformly and without shock. The moving head of the testing machine shall travel at the rate of approximately 0.05 in. per min. when the machine is running idle.

28. *Calculation of Compressive Strength.*—The total load indicated by the testing machine at failure of the test specimen shall be recorded, and the unit compressive strength calculated in pounds per square inch of the cross-sectional area of the specimen. Specimens that are manifestly faulty, or that give strengths differing by more than 15 per cent from the average value of all test specimens made from the same sample and tested at the same period, shall not be considered in determining the compressive strength.

Standard Specifications for

QUICKLIME AND HYDRATED LIME FOR COOKING OF RAGS IN PAPER MANUFACTURE[1]

A.S.T.M. Designation: C 45 - 25

ADOPTED, 1925.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 45; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover quicklime and hydrated lime suitable for use in the cooking of rags for the manufacture of paper.

NOTE.—Rags are cooked for the manufacture of paper in a digester under steam pressure with lime or with lime and soda ash. They are then washed to eliminate as much of the noncellulose material as possible.

General Requirements

2. (*a*) Either quicklime or hydrated lime may be furnished, as specified.

(*b*) The lime shall be clean and free from gritty substances.

Chemical Composition

3. The standard composition for quicklime and hydrated lime (percentages of available lime, calculated on the basis of the weight of sample taken at the place of manufacture) shall be as follows (Note):

	Quicklime	Hydrated Lime
Available lime, per cent...	90	64.3[a]

[a] Available lime of 64.3 per cent is equivalent to 85 per cent of available calcium hydroxide.

NOTE.—A standard composition without rejection limits is specified for the reason that lime of either higher or lower total oxides, available lime, calcium oxide, or calcium hydroxide than the standard may safely be used under suitable conditions for the purpose herein specified, depending primarily upon purely economic considerations. In the present state of the art, it is believed that the more serviceable type of specification for the product herein specified is that which defines a reasonable standard rather than one that fixes actual rejection limits. It is generally recognized that, other things being equal, lime meeting this standard is preferable to lime not meeting it, and lime surpassing the standard should be considered at a premium.

Methods of Testing

4. The chemical analysis for available lime shall be made in accordance with the Standard Methods of Chemical Analysis of Limestone, Quicklime, and Hydrated Lime (A.S.T.M. Designation:

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-7 on Lime.

[2] Prior to adoption as standard, these specifications were published as tentative from 1922 to 1925, being revised in 1924 and 1925. Editorially revised and rearranged in 1939.

C 25) of the American Society for Testing Materials.[3]

Sampling, Inspection, etc.

5. The sampling, inspection, rejection, retesting, packing, and marking shall be conducted in accordance with the Standard Methods of Sampling, Inspection, Packing, and Marking of Quicklime and Lime Products (A.S.T.M. Designation: C 50) of the American Society for Testing Materials.[3]

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for

QUICKLIME FOR SULFITE PULP MANUFACTURE[1]

A.S.T.M. Designation: C 46 – 27

ADOPTED, 1927.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 46; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover quicklime suitable for use in the manufacture of sulfite pulp.

NOTE.—Lime is used in the "Milk of Lime" or "Tank System" of sulfite pulp manufacture for making the cooking liquor. The milk of lime is held in solution or suspension in a series of tanks equipped with suitable agitators. The sulfur dioxide is forced or drawn through these tanks successively. In some cases, the tanks are built on top of each other in the form of a tower. The contents of the first tank are drawn off when the liquor has reached a certain strength (3.5 to 6 per cent total SO_2) and the contents of the second and third tanks progress to the first and second tanks respectively. The third tank is again charged with fresh milk of lime. There are other systems of absorption which provide for continuous instead of intermittent operation. The function of the lime is to furnish the base for the formation of the bisulfites of calcium and magnesium.

General Requirements

2. Either calcium or magnesium lime may be furnished, but owing to the greater solubility and reactivity of magnesium bisulfite, high-magnesium lime gives the best results. The ratio of MgO to CaO shall be reasonably constant, and the lime shall be moderately free from impurities.

Chemical Composition

3. The quicklime shall conform to the following requirements as to chemical composition, all percentages to be calculated on the basis of the weight of the sample taken at the place of manufacture:[a]

	Calcium Lime	Magnesium Lime
Calcium oxide, min., per cent	92.5	55.4
Magnesium oxide, per cent	2.5 max.	39.6 min.
Oxides of silicon, iron and aluminum, max., per cent	3.0	3.0

[a] If it is desired to use the nonvolatile basis the figures given above should be changed to the following:

	Calcium Lime	Magnesium Lime
Calcium oxide, min., per cent	94.3	56.5
Magnesium oxide, per cent	2.6 max.	40.4 min.
Oxides of silicon, iron and aluminum, max., per cent	3.1	3.1

Methods of Testing

4. The chemical analysis of the lime shall be made in accordance with the

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-7 on Lime.

[2] Prior to adoption as standard, these specifications were published as tentative from 1922 to 1927, being revised in 1924, 1925, and 1927. Editorially revised and rearranged in 1939.

Standard Methods of Chemical Analysis of Limestone, Quicklime, and Hydrated Lime (A.S.T.M. Designation: C 25) of the American Society for Testing Materials.[3]

Sampling, Inspection, etc.

5. The sampling, inspection, rejection, retesting, packing, and marking shall be conducted in accordance with the Standard Methods of Sampling, Inspection, Packing, and Marking of Quicklime and Lime Products (A.S.T.M. Designation: C 50) of the American Society for Testing Materials.[3]

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for

HYDRATED LIME FOR VARNISH MANUFACTURE[1]

A.S.T.M. Designation: C 47 - 27

ADOPTED, 1927.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 47; the final number indicates the year of original adoption as standard or, in the case of revision the year of last revision.

Scope

1. These specifications cover hydrated lime suitable for use in the manufacture of oleoresinous varnishes.

NOTE.—Hydrated lime is used in the manufacture of oleoresinous varnishes to harden and partially neutralize the rosin. About 8 lb. of hydrated lime are gradually added to 100 lb. of the melted rosin and heated for a short period.

General Requirements

2. (*a*) The hydrated lime shall be of such a color that no pronounced darkening of the finished varnish will result. It shall be of such a texture and of such a fineness that all portions of it will rapidly come into intimate contact with the resins so as to enter into the chemical reactions with rapidity.

(*b*) The lime shall be completely hydrated (Note), since a high degree of hydration is important.

NOTE.—A very high calcium lime is preferred. It is especially important that the iron content be low in order to avoid undesirable color effects.

Chemical Composition

3. The hydrated lime shall conform to the following requirements as to chemical composition, calculated to the non-volatile basis:

Calcium oxide (CaO), min., per cent	94.0
Magnesium oxide (MgO), max., per cent	3.0
Iron oxide, aluminum oxide, and insoluble matter, max., per cent	3.0
Iron oxide (Fe_2O_3), max., per cent	0.4
Carbon dioxide (CO_2), max., per cent:	
If sample is taken at place of manufacture	3.0
If sample is taken at other than place of manufacture	5.0

Fineness

4. Not more than 10 per cent of the hydrated lime shall be retained on a No. 230 (62-micron) sieve.

Reactivity

5. The hydrated lime shall conform to the requirements of the tung-oil heat test as indicated in Section 6 (*c*).

Methods of Testing

6. (*a*) *Chemical Analysis.*—The chemical analysis of the lime shall be made in accordance with the Standard Meth-

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-7 on Lime.

[2] Prior to adoption as standard, these specifications were published as tentative from 1922 to 1927. Editorially revised and rearranged in 1939.

ods of Chemical Analysis of Limestone, Quicklime, and Hydrated Lime (A.S.T.M. Designation: C 25) of the American Society for Testing Materials.[3]

(*b*) *Fineness.*—One hundred grams of the sample as received shall be placed on a No. 230 (62-micron) sieve.[4] The material shall be washed by means of a stream of water from a faucet (Note). Washing shall be continued until the water coming through the sieve is clear. The residue upon the No. 230 (62-micron) sieve shall be dried to constant weight in an atmosphere free from CO_2 in a drying oven whose temperature is maintained between 100 and 120 C. (212 and 248 F.). The weight of this residue shall be calculated as a percentage of the original sample.

NOTE.—A small piece of rubber tubing attached to a water faucet will be found convenient. The velocity of the stream of water may be increased by pinching the tube, but the velocity should not be sufficient to cause any danger of splashing the sample over the sides of the sieve.

(*c*) *Tung-Oil Heat Test.*—A 1-g. sample of hydrated lime shall be stirred into 50 g. of raw tung oil[5] in a 100-ml. beaker. The resulting cloudy mass shall be heated. High-quality hydrated lime will usually start to react at approximately 80 C. (176 F.), small bubbles rising to the surface. As heating is continued, large flakes of lime tungate will form and rise to the surface to form a thick white scum, and the oil below will gradually become fairly clear. These changes will usually be completed when a temperature of 120 C. (248 F.) is reached. The heating shall be continued until the mass reaches a temperature of 140 C. (284 F.). It shall then be allowed to cool and the formation noted of the thick scum on the surface which will, when freed of air, gradually drop to the bottom of the beaker. This scum consists of tung oil soaps of lime which are quite insoluble in the oil. Addition of rosin will readily effect solution of these soaps.

A poor grade of lime will fail to react, the absence of bubbles or flakes of lime tungate being noticeable. The oil will remain quiescent and cloudy. Only small amounts of soaps will form on the surface.

Sampling, Inspection, etc.

7. The sampling, inspection, rejection, retesting, packing, and marking shall be conducted in accordance with the Standard Methods of Sampling, Inspection, Packing, and Marking of Quicklime and Lime Products (A.S.T.M. Designation: C 50) of the American Society for Testing Materials.[3]

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

[4] Detailed specifications for this sieve are given in the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

[5] Tung oil for this test should conform to the Standard Specifications for Raw Tung Oil (A.S.T.M. Designation: D 12), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for

QUICKLIME AND HYDRATED LIME FOR USE IN THE TEXTILE INDUSTRY[1]

A.S.T.M. Designation: C 48 - 24

ADOPTED, 1924.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 48; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover quicklime and hydrated lime suitable for use in the textile industry in the treatment of the vegetable fibers (cotton, linen, and jute).

NOTE 1.—Lime is used in the textile industry for the following operations:

(*a*) Liming the kiers,
(*b*) Boiling out or scouring,
(*c*) Bleaching,
(*d*) Mercerizing,[3] and
(*e*) Water Softening.

These specifications apply only to the first three operations in connection with the treatment of the vegetable fibers: cotton, linen and jute. Lime for water softening is covered by the Standard Specifications for Quicklime and Hydrated Lime for Water Treatment (A.S.T.M. Designation: C 53) of the American Society for Testing Materials.[4]

NOTE 2.—Kier liming consists of applying a coating of whitewash to the inside of the kier and heating with steam. These two operations are repeated until a coating of the desired thickness is obtained. The lime serves to prevent the fibers from being stained by the iron kier.

NOTE 3.—The boiling-out process consists of soaking the fibers in a milk of lime, passing them through rollers into a kier, covering the fibers with water and boiling with steam either at atmospheric or higher pressures. The fibers then pass through a series of washings and treatments with acid and soda ash. The function of the lime is to saponify, hydrolyze, and neutralize whereby it makes possible, in following operations, the removal of fatty and greasy matters and pectin bodies. The fibers then consist of practically pure cellulose, the impurities being coloring matter not decomposed by alkalies.

NOTE 4.—Lime is used in the bleaching operation for making the "chemic" solution or hypochlorite bath. This bath may be made from bleaching powder or from lime and liquid chlorine. These specifications are limited to the process involving the use of lime and liquid chlorine. The operation consists of making a milk of lime and introducing chlorine under carefully controlled conditions until the mixture is nearly neutral. The sludge is allowed to settle and the clear hypochlorite solution is drawn off. After bleaching, the fibers are scoured and washed, and then passed on to the soda ash boil. Lime serves as a carrier of chlorine under conditions whereby the liberation of nascent oxygen and the resulting oxidation can be controlled in accordance with the special requirements of textile fibers. Bleach-

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-7 on Lime.

[2] Prior to adoption as standard, these specifications were published as tentative from 1922 to 1924. Editorially revised and rearranged in 1939.

[3] Work is in progress on separate specifications for causticization (which is involved in mercerization).

[4] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

ing decolorizes the fibers by oxidation of the coloring matter. It probably performs several other functions also, about which there is no general agreement.

General Requirements

2. Either quicklime or hydrated lime of fairly high calcium content may be furnished, as specified (Note).

NOTE.—There is a tendency to prefer quicklime for the boiling out and kier liming process, and hydrated lime for the mordanting process. Quicklime or hydrated lime which contains iron in a readily soluble form should be avoided as should also hydrated lime which contains a considerable amount of unhydrated magnesia.

Chemical Composition

3. The quicklime and hydrated lime shall conform to the following requirements as to chemical composition, calculated to the nonvolatile basis:

Calcium oxide (CaO), min., per cent	94.0
Magnesium oxide (MgO), max., per cent	3.0
Iron and aluminum oxides (Fe_2O_3 and Al_2O_3), max., per cent	2.0
Silica (SiO_2) and insoluble matter, max., per cent	2.5
Carbon dioxide (CO_2), max., per cent:	
If sample is taken at place of manufacture:	
Quicklime	3.0
Hydrated lime	5.0
If sample is taken at other than place of manufacture:	
Quicklime	5.0
Hydrated lime	7.0

Methods of Testing

4. The chemical analysis of the lime shall be made in accordance with the Standard Methods of Chemical Analysis of Limestone, Quicklime, and Hydrated Lime (A.S.T.M. Designation: C 25) of the American Society for Testing Materials.[4]

Sampling, Inspection, etc.

5. The sampling, inspection, rejection, retesting, packing, and marking shall be conducted in accordance with the Standard Methods of Sampling, Inspection, Packing, and Marking of Quicklime and Lime Products (A.S.T.M. Designation: C 50) of the American Society for Testing Materials.[4]

Standard Specifications for

QUICKLIME AND HYDRATED LIME FOR SILICA BRICK MANUFACTURE[1]

A.S.T.M. Designation: C 49 - 42

ADOPTED, 1924; REVISED, 1942.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 49; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover quicklime and hydrated lime suitable for use in the manufacture of silica brick (Note).

NOTE.—In the manufacture of silica brick, silica in the form of massive quartzite or quartz conglomerate is ground until the particles are less than ½ in. in size. Lime in the form of either slaked lime or hydrated lime is then added in quantities varying from 1.5 to 3.0 per cent CaO and the shapes are molded and dried. They are then burned in down-draft or tunnel kilns until most of the quartzite has been converted into tridymite or cristobalite.

General Requirements

2. Either quicklime or hydrated lime may be used. The lime shall be substantially free from ash, dirt, and core. Fineness of hydrate and rapidity of slaking of quicklime are desirable qualities.

Chemical Composition

3. The quicklime or hydrated lime shall conform to the following requirements as to chemical composition, calculated to the nonvolatile basis:

Calcium oxide (CaO), min., per cent	90.0
Magnesium oxide (MgO), max., per cent	4.5
Combined iron and aluminum oxides (Fe_2O_3 and Al_2O_3), max., per cent	1.5
Silica (SiO_2) and insoluble matter, max., per cent	3.0
Carbon dioxide (CO_2), max., per cent:	
If sample is taken at place of manufacture	5.0
If sample is taken at other than place of manufacture	10.0

Methods of Testing

4. The chemical analysis of the lime shall be made in accordance with the Standard Methods of Chemical Analysis of Limestone, Quicklime, and Hydrated Lime (A.S.T.M. Designation: C 25) of the American Society for Testing Materials.[3]

Sampling, Inspection, etc.

5. The sampling, inspection, rejection, retesting, packing, and marking shall be conducted in accordance with the Standard Methods of Sampling, Inspection, Packing, and Marking of Quicklime and Lime Products (A.S.T.M. Designation: C 50) of the American Society for Testing Materials.[3]

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-7 on Lime.

[2] Prior to adoption as standard, these specifications were published as tentative from 1922 to 1924.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for

QUICKLIME AND HYDRATED LIME FOR WATER TREATMENT[1]

A.S.T.M. Designation: C 53 – 39

ADOPTED, 1927; REVISED, 1939.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 53; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover lime suitable for use in the purification and softening of water for municipal and industrial supplies.

NOTE.—In the treatment of water for municipal and industrial supplies, lime is used alone or with iron sulfate or aluminum sulfate to produce a precipitate which assists in the clarification of the water and in the removal of the bacteria by filtration. An excess of lime is sometimes used to remove part of the hardness of the water. Lime and soda ash are used together for softening water. The lime serves as a chemical reagent in water treatment. The only useful constituent of the lime is the calcium oxide content of the quicklime or hydrated lime capable of reacting with the other chemicals in the water or added to it. Inert material, besides reducing the value in proportion to its amount, also makes more sludge to be disposed of for a given amount of chemical action and thus reduces the capacity of the equipment in which it is used.

General Requirements

2. (*a*) Either quicklime or hydrated lime may be furnished, as specified.

(*b*) Quicklime shall be substantially free of core, ash, and dirt, and shall be capable of disintegrating in water into a suspension of finely divided material.

Chemical Composition

3. The standard composition for quicklime and hydrated lime (percentages of available lime, calculated on the basis of the weight of sample taken at the place of manufacture) shall be as follows (Note):

	Quicklime	Hydrated Lime
Available lime, per cent . .	90	68.1[a]

[a] Available lime of 68.1 per cent is equivalent to 90 per cent of calcium hydroxide.

NOTE.—A standard composition without rejection limits is specified for the reason that lime of either higher or lower total oxides, available lime, calcium oxide, or calcium hydroxide than the standard may safely be used under suitable conditions for the purpose herein specified, depending primarily upon purely economic considerations. In the present state of the art, it is believed that the more serviceable type of specification for the product herein specified is that which defines a reasonable standard rather than one that fixes actual rejection limits. It is generally recognized that, other things being equal, lime meeting this standard is preferable to lime not meeting it, and lime surpassing the standard should be considered at a premium.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-7 on Lime.

[2] Prior to adoption as standard, these specifications were published as tentative from 1924 to 1927, being revised in 1925 and 1927.

These specifications comprise a revision and consolidation of the former Standard Specifications for Quicklime for Use in Water Treatment (C 53 – 27) and for Hydrated Lime for Use in Water Treatment (C 54 – 27).

Methods of Test

4. The chemical analysis for available lime shall be made in accordance with the Standard Methods of Chemical Analysis of Limestone, Quicklime, and Hydrated Lime (A.S.T.M. Designation: C 25) of the American Society for Testing Materials.[3]

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Sampling, Inspection, etc.

5. The sampling, inspection, rejection, retesting, packing, and marking shall be conducted in accordance with the Standard Methods of Sampling, Inspection, Packing, and Marking of Quicklime and Lime Products (A.S.T.M. Designation: C 50) of the American Society for Testing Materials.[3]

Standard Specifications for

SAND FOR USE IN PLASTER[1]

A.S.T.M. Designation: C 35 – 39

ADOPTED, 1939.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 35; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Quality of Sand.—It is recognized that for certain purposes satisfactory results may be obtained with sands not conforming to these specifications. In such cases the use of sand not conforming to these specifications may be authorized only under special provisions based upon laboratory studies of the possibility of designing a mixture of materials to be used on the job that will yield plaster equivalent in quality to the specified mixture made with sand complying with these specifications in all respects.

Definition

1. Sand[3] for use in plaster shall consist of fine granular material composed of hard, strong, durable, uncoated particles which are free from injurious amounts of saline, alkaline, organic, or other deleterious substances.

Grading (Sieve Analysis)

2. (*a*) Sand for use in plaster shall be uniformly graded from fine to coarse within the following limits:

Retained on Sieve	Percentage Retained Max.	Min.
No. 4 (4760-micron)	0	..
No. 8 (2380-micron)	10	0
No. 30 (590-micron)	80	15
No. 50 (297-micron)	95	70
No. 100 (149-micron)	..	95

(*b*) The amount of material finer than a No. 200 (74-micron) sieve, shall not exceed 5 per cent.

(*c*) The sieves shall conform to the requirements of the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials.[4]

Organic Impurities

3. The sand when subjected to the colorimetric test for organic impurities shall show a color not darker than the standard color, unless it is shown by adequate tests that the impurities causing the color are not harmful in plaster.

[1] Under the standardization procedure of the Society, these specifications are under the joint jurisdiction of the A.S.T.M. Committee C-7 on Lime and Committee C-11 on Gypsum.

[2] Prior to their present adoption as standard, these specifications were published as tentative from 1921 to 1925, being revised in 1924 and 1925. They were adopted in 1925, revised in 1930, but withdrawn and republished as tentative in 1936.

These specifications are in effect a revision and consolidation of, and replace the former Standard Specifications for Sand for Use in Gypsum Plaster (A.S.T.M. Designation: C 35) and the Tentative Specifications for Sand for Use in Lime Plaster (A.S.T.M. Designation: C 66 – 31 T), which specifications were accordingly discontinued in 1936.

[3] The term sand is defined in the Standard Definition of the Term Sand (A.S.T.M. Designation: C 58), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

[4] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Methods of Testing

4. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials:

(*a*) *Sieve Analysis.*—Standard Method of Test for Sieve Analysis of Fine and Coarse Aggregates (A.S.T.M. Designation: C 136).[4]

(*b*) *Material Finer than No. 200 Sieve.*—Standard Method of Test for Amount of Material Finer than No. 200 Sieve in Aggregates (A.S.T.M. Designation: C 117).[4]

(*c*) *Organic Impurities.*—Standard Method of Test for Organic Impurities in Sands for Concrete (A.S.T.M. Designation: C 40).[4]

Standard Methods of

SAMPLING, INSPECTION, PACKING, AND MARKING OF QUICKLIME AND LIME PRODUCTS[1]

A.S.T.M. Designation: C 50 - 27

ADOPTED, 1927.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 50; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods outline procedures for the sampling, inspection, rejection, retesting, packing, and marking of quicklime and lime products and are intended for use in connection with the individual specifications for lime.

SAMPLING

General

2. (*a*) Samples of quicklime and lime products shall be taken at the place of manufacture or at the destination, as may be agreed upon by the seller and the purchaser. If the samples are taken elsewhere than at the place of manufacture of the material, such samples shall be taken within 24 hr. of the receipt of the material.

(*b*) Samples may be taken from conveyors delivering to bins or piles, from filled bins by means of sampling tubes, from filled bins at the point of discharge, from piles by means of sampling tubes or shovels, or from loaded cars.

(*c*) Sampling shall be conducted as expeditiously as possible in order to avoid undue exposure of the material to the air. Samples shall not be taken from broken packages.

(*d*) Samples shall be taken in triplicate and immediately sealed in airtight, moistureproof containers. Samples shall be of an equal weight of not less than 5 lb. each when the quantity sampled equals 30 tons or more. For smaller quantities, the weights of the samples may be reduced proportionately, provided that no sample shall weigh less than 2 lb., except by agreement between the manufacturer and the purchaser.

(*e*) One sample shall be immediately delivered to the consignee; one sample shall be delivered to the consignor, if requested, within ten days from the date of sampling; the third sample shall be retained with the seal unbroken until such time as the material has been accepted or a referee test called for.

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee C-7 on Lime.

[2] Prior to adoption as standard, these methods were published as tentative from 1922 to 1927, being revised in 1923, 1924, and 1927. Editorially revised and rearranged in 1939.

Lump or Granular Material in Bulk

3. (*a*) *Piles or Cars.*—Material shall be selected so that it will represent an average of all parts of the pile or car and shall not contain a disproportionate share of the top or bottom layers. This material shall comprise at least ten shovelfuls taken from different parts of the pile or car. The total quantity so taken shall weigh not less than 100 lb. for a unit of 30 tons of material, with a proportionate quantity for multiples of this unit, provided that at no time shall the total amount of material so taken weigh less than 50 lb. It shall be broken, if necessary, to pass a 1-in. ring, thoroughly mixed and quartered, and the triplicate samples for testing shall be taken.

(*b*) *At Conveyors to or from Bins, Piles, or Cars.*—Material shall be taken at regular intervals during the entire time of movement of the mass being sampled. The quantity to be taken at each interval and the number of such intervals shall be so proportioned that the final quantity of material taken shall conform to the requirements prescribed in Paragraph (*a*).

Lump or Granular Material in Packages

4. At least 2 per cent of the packages shall be sampled, but in no case shall less than five packages be sampled. Individual packages shall be taken from various parts of the unit being sampled. The contents of each of the packages so taken shall be put in a pile, broken, if necessary, to pass a 1-in. ring, thoroughly mixed and quartered, and the triplicate samples for testing shall be taken.

Powdered Material in Bulk

5. (*a*) *Bins, Piles, or Cars.*—A sampling tube shall be used which takes a core not less than 1 in. in diameter, and which is of sufficient length to permit the taking of a core from the top to the bottom of the mass being sampled. Cores shall be taken from at least ten separate parts of the mass. The cores so taken shall be thoroughly mixed and quartered, and the triplicate samples for testing shall be taken from the material so selected and prepared. A minimum quantity of 100 lb. shall be taken for a unit of 30 tons of material, with a proportionate quantity for multiples of this unit, provided that at no time shall less than 50 lb. be taken as the original portion.

(*b*) *At Conveyors to or from Bins, Piles, or Cars, at Packing Machines, etc.*—Sampling at conveyors, packing machines, etc., shall conform to the provisions of Section 3 (*b*).

Powdered Material in Packages

6. At least 1.5 per cent of the packages shall be sampled, but in no case shall less than five packages be sampled. Individual packages shall be taken from various parts of the unit being sampled. A sampling tube shall be used which takes a core not less than $\frac{1}{2}$ in. in diameter. The tube shall be inserted into the package being sampled so that it will take a core of the material from substantially the entire length of the package. Material thus taken from individual packages shall be thoroughly mixed and quartered, and the triplicate samples for testing shall be taken from the material so selected and prepared.

Treatment of Sample at Laboratory

7. (*a*) *Sample for Chemical Analysis.*—The sample as received at the laboratory shall be thoroughly mixed, and reduced in quantity by the usual methods of quartering to give a minimum portion of 75 g. This portion shall be ground, either by a suitable mechanical device or by hand in an agate mortar, until it all passes a No. 100 (149-micron)

sieve.[3] This 75-g. sample shall then be thoroughly mixed and placed in an airtight container for use in the chemical analysis.

(*b*) *Samples for Special Tests.*—Samples to be used for such special tests as may be required by individual specifications shall be obtained by mixing and quartering the material remaining after removal of the 75-g. portion for chemical analysis. The quantity of sample and the sieve size of the particles will depend upon the requirements of the individual specifications.

INSPECTION

Inspection

8. When inspection and sampling are to be conducted at the place of manufacture, the manufacturer shall afford the inspector representing the purchaser all reasonable facilities for inspection and sampling, which shall be so conducted as not to interfere unnecessarily with the operation of the works. The duties of the inspector or sampler shall be confined exclusively to sampling and inspection of the finished material as ready for shipment, and shall not under any circumstances be construed to permit investigation of methods or processes of manufacture.

REJECTION

Rejection

9. (*a*) Rejection of material based on failure to pass the tests prescribed in the specifications shall be reported to the manufacturer within ten days from the taking of samples, and the cause for rejection shall be stated.

(*b*) The samples which represent rejected material shall be kept in airtight, moistureproof containers for at least five days from the date of the original test report.

RETESTING

Retesting

10. (*a*) Either of the contracting parties may make claim for a retest within five days of the date of the original test report. The expense of the retest shall be borne by the party demanding such retest.

(*b*) Should the contracting parties be unable to reach a mutually satisfactory agreement based upon the results of the original test, the third sample of the material shall be delivered unopened to a referee laboratory for test and the results of this referee test shall be binding on both parties.

PACKING

Packing

11. (*a*) *Lump Lime.*—Lump lime may be shipped in bulk in carload lots, in wooden barrels or steel containers holding 180 lb. or 280 lb. net each, or in pasteboard cartons holding 90 lb. net each.

(*b*) *Pulverized Lime.*—Pulverized lime may be shipped in bulk, in carload lots, or in cloth or paper bags.

(*c*) *Hydrated Lime.*—Hydrated lime shall be shipped in cloth bags holding 100 lb. net each, or in paper bags holding 50 lb. net each.

MARKING

Marking

12. (*a*) Each shipment of material shall carry with it some means of identification, stating the particular contract on which the purchase and shipment were made.

(*b*) Each package shall have legibly marked thereon, the net weight of its contents, the name of the manufacturer, the place of manufacture, and the brand

[3] Detailed requirements for this sieve are given in the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

name, if any. This marking shall comply with all requirements of the Federal Lime Barrel Act, entitled "An Act to Standardize Lime Barrels (Public No. 228 - Congress - S 5425)," and with such other federal or state laws as may be applicable to the shipment in question.

(*c*) In addition to the above information, the following guarantee may be marked on each package or shipment: "Guaranteed by (name of manufacturer) to meet the specifications of the American Society for Testing Materials" for (name of material).

Standard Methods of

CHEMICAL ANALYSIS OF LIMESTONE, QUICKLIME, AND HYDRATED LIME[1]

A.S.T.M. Designation: C 25 - 44

ADOPTED, 1929; REVISED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 25; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods of test cover procedures for the chemical analysis of limestone, quicklime, and hydrated lime.

NOTE.—"The Analysis of Silicate and Carbonate Rocks," U. S. Geological Survey *Bulletin 700*, should be used for reference in connection with these methods, especially when very exact and detailed analyses are desired. Blank determinations should be made on all reagents, and corrections applied when necessary.

Samples for Analysis

2. Samples for chemical analysis shall be taken and prepared in accordance with the requirements of the Standard Methods of Sampling, Inspection, Packing, and Marking of Quicklime and Lime Products (A.S.T.M. Designation: C 50) of the American Society for Testing Materials[3] applicable to the material to be tested.

Special Solution Required

3. *Standard Potassium Permanganate Solution.*—Dissolve 5.64 g. of $KMnO_4$ in 1000 ml. of water. Let age for one week or more. Filter through purified asbestos and standardize against the National Bureau of Standards' standard sample 40c of sodium oxalate as follows: Transfer 0.5 g. of the standard sodium oxalate, dried at 105 C., to a 400-ml. beaker. Add 250 ml. of diluted H_2SO_4 (5:95), freshly boiled for 10 to 15 min. and cooled to 27 ± 3 C. Stir until the oxalate has dissolved. Add 40 to 42 ml. of the standard $KMnO_4$ at the rate of 25 to 35 ml. per min., while stirring slowly. Let stand until the pink color disappears (about 60 sec.). (If the pink color should persist because of the addition of an excess of $KMnO_4$ solution, discard and begin again, adding a few milliliters less of the $KMnO_4$.) Heat to 55 to 60 C. and complete the titration at this temperature by adding $KMnO_4$ until a faint pink color persists for 30 sec. Add the last 0.5 to 1 ml. drop by drop, with particular care to allow each drop to become decolorized

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee C-7 on Lime.

[2] Prior to adoption as standard, these methods were published as tentative from 1919 to 1929, being revised in 1921, 1922, 1925, 1926, and 1927.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

before the next is introduced. Determine the excess of $KMnO_4$ required to impart a pink color to the solution by matching the color in another beaker containing the same amount of the specially treated diluted H_2SO_4 at 55 to 60 C. One milliliter of the standard $KMnO_4$ will be approximately equivalent to 0.005 g. of CaO.[4] The exact value shall be calculated from the following equations:

$$5\ Na_2C_2O_4 + 2\ KMnO_4 + 8\ H_2SO_4 = 5\ Na_2SO_4 + K_2SO_4 + 2\ MnSO_4 + 10\ CO_2 + 8\ H_2O$$

$$5\ CaC_2O_4 + 2\ KMnO_4 + 8\ H_2SO_4 = 5\ CaSO_4 + K_2SO_4 + 2\ MnSO_4 + 10\ CO_2 + 8\ H_2O$$

Insoluble Matter Including Silicon Dioxide

4. Weigh out approximately 0.5 g. of the sample[5] and, if a limestone or hydrated lime, ignite in a covered platinum crucible in an electric muffle[6] for 15 min., or longer if the heat is not powerful enough to effect complete decomposition within that time. Transfer to an evaporating dish, preferably of platinum[7] for the sake of celerity in evaporation, mix to a thin slurry with distilled water, add 5 to 10 ml. of HCl (sp. gr. 1.18), and digest with aid of gentle heat and agitation until solution is complete. Solution may be aided and the time shortened by light pressure with the flattened end of a glass rod upon resistant lumps. Then evaporate the solution to dryness, so far as this may be possible, on the water bath. When dry or nearly so, place the dish and its contents (covered) in an air bath or on a platinum triangle resting on a hot plate and, if a limestone of high calcium content is under treatment, heat for 1 hr. at 200 C.; if a limestone of high magnesium content is under treatment the temperature should not exceed 120 C. Drench the cooled mass with HCl (sp. gr. 1.18) and allow to stand for a few minutes. Add an equal volume of water, cover the dish, and place on the water bath for 10 min.[8] Separate the SiO_2 by filtration on a filter of suitable size. Wash thoroughly with diluted HCl and then wash twice with cold water. Evaporate the filtrate to dryness. Extract the residue with HCl as before but allowing only a few minutes time. Filter this solution through a second and smaller paper. Transfer the wet papers containing the separated residue to a weighed platinum crucible. Char carefully without allowing the paper to inflame, and then ignite to constant weight in an electric muffle.[6] The increase in weight represents the insoluble and siliceous matter and is reported as insoluble matter, including silicon dioxide.

Silicon Dioxide

5. (*a*) Treat the "insoluble matter including silicon dioxide," (Section 4) in the crucible with 5 ml. of water, 5 ml. of HF and one or two drops of H_2SO_4,[9] and evaporate to dryness. Heat the residue for 2 or 3 min. in an electric muffle[6] and again weigh. The difference between this weight and that previously obtained gives the weight of SiO_2.[10]

[4] With high calcium limes, more than 50 ml. of this solution probably will be required to titrate the liberated oxalic acid. In such cases the use of a larger burette is to be preferred to increasing the strength of the permanganate solution.

[5] Due to the rapidity with which quicklime and hydrated limes become contaminated by absorption of water and carbon dioxide from the air, samples must be protected in tightly stoppered containers at all times. Samples for analysis are to be weighed from stoppered weighing bottles, the exact weight of the samples being determined by the difference in weight of the bottles before and after the samples are removed.

[6] Ignition of the sample in an electric muffle is far superior to flame ignition. If an electric muffle is not available, flame ignition and the blast lamp may be used.

[7] If a platinum dish is not available, porcelain may be used. A glass container positively must *not* be used.

[8] Instead of strong acid, that of half strength may be used and the heating begun at once.

[9] See U. S. Geological Survey *Bulletin 700*, p. 103.

[10] For ordinary control work in the plant laboratory this correction may, perhaps, be neglected; the double evaporation must never be neglected.

(*b*) To determine SiO_2 by volatilization with HF, fuse the residue in the crucible with a little Na_2CO_3,[11] cool the melt and dissolve it in diluted HCl. Add the solution to the filtrate from the second evaporation for "insoluble matter including silicon dioxide" (Section 4). If the "insoluble matter including silicon dioxide," is reported as such and no HF treatment is given, the "insoluble matter including silicon dioxide," is not fused or further treated.

Insoluble Matter other than Silicon Dioxide

6. The difference between the weight of "insoluble matter including silicon dioxide" and silicon dioxide represents the "insoluble matter other than silicon dioxide."

Iron and Aluminum Oxides

7. To the HCl solution from the determination of SiO_2 add a few drops of bromine water or HNO_3 and boil the solution until all trace of bromine or chlorine is gone. Then add HCl, if not already present, sufficient to ensure a total volume of 10 to 15 ml. of HCl (sp. gr. 1.18).[12] Add a few drops of methyl red solution and after diluting to 200 to 250 ml. heat the liquid to boiling. Neutralize by means of NH_4OH (diluted towards the end) until the color of the liquid changes to a distinct yellow. Boil for 1 or 2 min., allow to settle, filter, and immediately wash the precipitate two or three times with hot NH_4Cl (2 per cent) and dry by suction.[13] Set the filtrate aside. Dissolve any precipitate from the paper in hot, diluted HCl, the solution passing into the beaker in which the precipitation was made, and wash the paper thoroughly with hot water. Boil the solution to expel any trace of chlorine, and precipitate the hydroxides with NH_4OH as before. Ignite the moist precipitate in a weighed platinum crucible,[7] cool, and weigh as Al_2O_3 and Fe_2O_3.[14]

Total Iron, Standard Procedure

8. Fuse the combined iron and aluminum oxides[15] (Section 7) in a platinum crucible at a very low temperature with 3 to 4 g. of $Na_2S_2O_7$ or $K_2S_2O_7$.[16] Treat the melt with sufficient diluted H_2SO_4 to insure the presence of not less than 5 g. of absolute acid and enough water to effect solution on heating. Evaporate the solution and eventually heat until acid fumes come off copiously. After cooling and redissolving in water, filter out the small amount of SiO_2, wash, ignite, weigh, and correct by HF and H_2SO_4.[17] Add the weight so corrected to the weight of SiO_2 previously found and deduct from the gross weight of the oxides of iron and aluminum. Reduce the filtrate by zinc.[18] Titrate with a 0.05 *N* $KMnO_4$ solution.

Total Iron, Alternative Procedure

9. (*a*) Dissolve 2 to 5 g. (depending upon the amount of iron present) of the properly prepared sample in HCl and evaporate rapidly to dryness. Treat the residue with water and HCl, filter off the SiO_2, and wash several times with hot water. Precipitate the iron in a

[11] Fusion with pyrosulfate is to be avoided on account of the subsequent introduction of sulfates into the solution.

[12] See U. S Geological Survey *Bulletin 700*, p. 258.

[13] See U. S. Geological Survey *Bulletin 700*, p. 107.

[14] This precipitate may also contain TiO_2, P_2O_5, and Mn_3O_4.

[15] When the iron is present in small quantities it is rather unsatisfactory to determine it in the ignited oxides from the 0.5-g. sample. Under these conditions the alternative procedure may be used.

[16] The pyrosulfates are less troublesome and more effective than the bisulfates.

[17] This correction for impurities should not be made when the HF correction of the main SiO_2 determination has been omitted. After two evaporations, 1 to 2 mg. of SiO_2 are still to be found with the R_2O_3 precipitate.

[18] Zinc will also reduce titanium. If this metal is to be determined and deducted from Al_2O_3, then H_2S must be used for reduction (boiling out the H_2S in a stream of CO_2 before titration). If titanium is not to be determined, the slight error introduced by its reduction with zinc is so small as to be negligible except in the most exact and detailed analyses.

boiling solution with fixed alkali, allow to settle, filter, and wash free of chlorides, using hot water. Dissolve in diluted H_2SO_4.

(*b*) Ignite the insoluble matter from the evaporated HCl solution in a platinum crucible. Treat with H_2SO_4 and HF and heat until fumes of H_2SO_4 appear. Bring the residue into solution with the addition of a few drops more of H_2SO_4 and combine the solution with that of the bulk of the iron. Reduce this solution by zinc and titrate the iron with 0.05 *N* $KMnO_4$.

Aluminum Oxide

10. Subtract the calculated weight of Fe_2O_3, obtained in accordance with Section 8, from the weight of $Al_2O_3 + Fe_2O_3$ (Section 7). Report the remainder as Al_2O_3. In case phosphorus is determined, calculate it to P_2O_5 and deduct also.

Calcium Oxide by Gravimetric Method[19]

11. Add a few drops of NH_4OH to the combined filtrate from the $Al_2O_3 + Fe_2O_3$ precipitate, and bring the solution to boiling. To the boiling solution add 35 ml. of a saturated solution of $(NH_4)_2C_2O_4$, and continue the boiling until the precipitated CaC_2O_4 assumes a granular form. Allow it to stand 20 min., or until the precipitate has settled and the supernatant liquid is clear, filter, and wash thoroughly with boiling water.[20] Place the wet filter and precipitate in a platinum crucible, and burn off the paper over the small flame of a bunsen burner or in a muffle. Ignite the residue, dissolve in hot diluted HCl, and make up the volume of the solution to 100 ml. with water. Add NH_4OH in slight excess and boil the liquid. If a small amount of $Al_2(OH)_6$ separates, filter it out, wash with NH_4Cl (2 per cent), ignite, weigh, and add this weight to that found in the first determination. Then reprecipitate the lime by $(NH_4)_2C_2O_4$, allow to stand until settled, filter, wash thoroughly with boiling water,[20] and weigh as CaO after ignition to constant weight in a covered platinum crucible.

Calcium Oxide by Volumetric Method[21]

12. Make the filtrate from the iron and aluminum oxide precipitate alkaline with NH_4OH, boil, and add 35 ml. of a boiling saturated solution of $(NH_4)_2C_2O_4$. Stir vigorously and allow to stand until the precipitate has settled, filter on an 11-cm. paper, and wash ten times with hot water.[22] Transfer the paper and precipitate to the beaker in which the precipitation was made, spreading the paper out against the upper portion of the beaker. Wash the precipitate from the paper with a jet of hot water, fold the paper, and leave it adhering to the upper portion of the beaker. Add to the contents of the beaker 50 ml. of diluted H_2SO_4 (1:10), dilute to a volume of 250 ml. with hot water, and heat to 80 to 90 C. Titrate with the standard $KMnO_4$ solution (Section 3) until the pink end point is obtained. Now drop the folded filter paper (which has been adhering to the side of the beaker) into the liquid; the pink color of the latter will be discharged. Finish the titration by adding $KMnO_4$ drop by drop until the pink end point is again obtained. A Gooch crucible may be used instead of filter paper. From the total quantity of standard $KMnO_4$ solution used, calculate the percentage of CaO.

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[19] The gravimetric method must be employed when a recovery of aluminum is desired, or when a determination of strontium is contemplated.

[20] Care must be exercised in this washing, as 1000 ml. of boiling water will dissolve over 10 mg. of CaC_2O_4.

[21] The volumetric method may be used for ordinary control work in the plant laboratory. It must be borne in mind that in this method all strontium oxide is reported as calcium oxide.

[22] Use not more than 125 ml. for this wash.

Strontium Oxide

13. Transfer the weighed oxides obtained in the gravimetric determination of calcium oxide (Section 11) to a small flask of 20-ml. capacity and dissolve in HNO_3. Evaporate to dryness and heat at 150 to 160 C. Treat the thoroughly dried nitrates with as little (rarely over 2 ml.) of a mixture of equal parts of absolute alcohol and ether as may be needed to dissolve the calcium salt, solution being hastened by occasional gentle agitation. After standing over night in the corked flask, collect the insoluble matter on the smallest possible filter and wash with more of the above mixture of alcohol and ether. After drying, pass a few milliliters of hot water through the filter, on which may remain a few tenths of a milligram of residue which usually does not contain any lime or other alkaline earth and whose weight is therefore to be deducted from that of the lime unless it can be shown that it is derived from the glass of the small flask in which the nitrates of calcium and strontium were evaporated. To the solution of strontium nitrate in a small beaker add a few drops of H_2SO_4 and then its volume of alcohol, whereby the strontium is precipitated as sulfate. After 12 hr., filter on a small filter paper, and wash the residue with 50 per cent alcohol. Ignite at a low temperature, moisten with diluted H_2SO_4, evaporate, again ignite, and weigh as $SrSO_4$. Test the sulfate spectroscopically as to freedom from calcium and barium. Calculate the weight of SrO and deduct from the weight of CaO as obtained in Section 11.

Magnesium Oxide

14. (*a*) Acidify the combined filtrates from the calcium precipitates with HCl, concentrate on the water bath to about 150 ml., and heat to boiling. To the boiling solution add 10 ml. of a saturated solution of $Na(NH_4)HPO_4$ and continue the boiling for several minutes. Then cool to room temperature, and add NH_4OH drop by drop, while stirring constantly, until the crystalline ammonium-magnesium orthophosphate begins to form and then in moderate excess, the stirring being continued for several minutes. Allow the liquid to stand in a cool atmosphere for from 12 to 48 hr.[23] and filter.

(*b*) Dissolve the precipitate in hot diluted HCl, dilute the solution with water to about 100 ml., add 1 ml. of the saturated solution of $Na(NH_4)HPO_4$ and NH_4OH drop by drop, while stirring constantly, until the precipitate is again formed as above described and the NH_4OH is present in moderate excess. Then allow it to stand in a cool atmosphere for from 12 to 48 hr.[23] Filter on paper or in a Gooch crucible, wash with diluted NH_4OH containing NH_4NO_3,[24] ignite,[6] cool, and weigh as $Mg_2P_2O_7$.[25]

Loss on Ignition

15. Place 1 g. of the prepared sample[26] in a weighed platinum crucible, cover with the lid, and heat gently[6] for 5 min., gradually increasing the temperature to the maximum of the muffle, and maintain at this temperature until constant weight is obtained (usually about 15 min.). The difference between the original weight of the sample and the final weight represents the loss on ignition.

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[23] The less the amount of magnesium present, the longer the precipitate must be allowed to settle.

[24] Made by diluting NH_4OH with distilled water until the solution contains 2.5 per cent of NH_3 by weight, then adding three or four drops of HNO_3 (sp. gr. 1.42).

[25] When manganese is present in the sample, it will be caught in large part with the magnesium precipitate. (See U. S. Geological Survey *Bulletin 700*, p. 134.) In case manganese was not eliminated before this precipitation, the weighed pyrophosphate must be dissolved in HNO_3, tested for manganese with bismuthate, the manganese determined and deducted as $Mn_2P_2O_7$.

[26] This determination may be made, if desired, upon the 0.5-g. sample ignited for solution in HCl as described in Section 4.

Mechanical Moisture in Limestone

16. Weigh 1 g. of the prepared sample in a tared, wide, low-form, flat-bottomed weighing bottle. Heat uncovered in a ventilated drying oven at 120 C. for 2 hr. Quickly stopper, cool in a desiccator, and weigh, lifting the stopper momentarily just before weighing. The use of a similar weighing bottle as a counterpoise carried through all of the operations is a desirable procedure. The loss in weight represents "mechanical moisture" or "hygroscopic water" at 120 C.

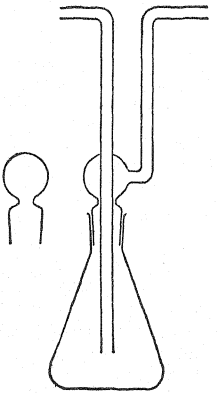


FIG. 1.—Bottle for Determination of Superficial Moisture.

Mechanical Moisture in Hydrated Lime

17. (*a*) *Arrangement of Apparatus.*—Mechanical moisture in hydrated limes shall be determined by aspirating over the sample in a closed container at 120 C. a slow stream of dry, CO_2-free air. The container for the sample shall be equipped with two interchangeable stoppers one to be used during weighings and the other to be used during the drying. The second stopper shall be equipped with two entry tubes for conducting the air stream over the sample.[27] (See Fig. 1.) The container shall be connected in a train, having before it a soda-lime tower, a lime-water bottle, a sulfuric acid bottle, and a phosphoric anhydride bulb, in the order named, and after it another protective phosphoric anhydride bulb. The lime-water bottle is useful for giving indication of the exhaustion of the soda lime. The train shall be placed in position, with connection for the sample bottle, in a drying oven at 120 C.

(*b*) *Procedure.*—Place 2.5 to 3 g. of the prepared sample into the previously weighed bottle, and immediately restopper it. A glazed paper funnel will aid rapid manipulation. Insert the bottle in the train by quickly exchanging the stoppers, and draw a slow current of dry, CO_2-free air through the apparatus for 2 hr. Remove the sample bottle from the oven with another quick exchange of stoppers, and set it in a desiccator to cool. When cool, remove it to the balance case for several minutes before weighing it, and just before weighing, lift the stopper slightly for an instant to relieve any vacuum that may exist in the bottle. The loss in weight of the sample represents "mechanical moisture" or "hygroscopic water" at 120 C. Use a bottle similar to the one containing the sample as a counterpoise in all weighings.

Carbon Dioxide by Standard Method[28]

18. (*a*) *Size of Sample.*—With limestone, use 0.5 g. of the sample; with

[27] A small bottle of 20 to 30-ml. capacity with glass stoppers, as described, ground to fit, is a convenient form of apparatus. (See Fig. 1.)

[28] This method is in accordance with the method given in U. S. Geological Survey *Bulletin 700*, p. 217. An illustration showing the arrangement of the component parts of the necessary apparatus is shown on p. 218 of that Bulletin.

burned lime, hydrated lime, etc., use 5 g. of the sample.

(*b*) *Procedure.*—Boil the weighed amount of prepared sample with diluted HCl(1:1) in a small Erlenmeyer flask attached to an upwardly inclined condenser, whence, after passing through a drying system—calcium chloride, anhydrous copper sulfate to retain hydrogen sulfide from decomposable sulfides and any HCl that may pass over, then calcium chloride again—the CO_2 is caught by absorption tubes filled with soda lime followed by calcium chloride. the observation bulbs (containing H_2SO_4 to show the rate of gas flow) being attached directly to the drying system. Then close the stopcock in the separatory funnel, half fill the latter with HCl (1:1), replace the rubber stopper of the funnel, insert the absorption tubes between the drying system and the observation bulbs, and allow the acid to flow into the flask, slowly if there is much CO_2, rapidly if there is but little. When effervescence diminishes in the former case, at once in the latter, light the burner under the flask and start the

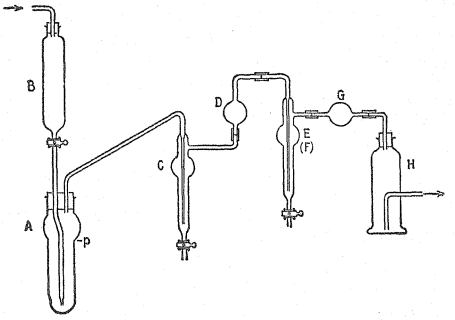


FIG. 2.—Apparatus for Determination of Carbon Residue.

Arrangements should be made for a current of air free from CO_2 with which to sweep out the apparatus before and after the determination, and for a slow current during its continuance. The results are very accurate and the determination can be quickly carried out. The manipulations are as follows: Pour hot water upon the powder in the flask fitted with a separatory funnel and delivery tube; attach this to the condenser, and force a current of air free from CO_2 through the whole system, except the weighed absorption tubes, until the original air has been displaced, flow of water through the condenser. Keep the flame low, so as to secure steady but quiet ebullition, and do not interrupt the air current although it should be reduced to a slow rate. With much CO_2 the rate of absorption is very readily noted by holding the hand to the soda-lime tubes, which become hot or warm when absorption is taking place. Sufficient time having elapsed,[29] extinguish the flame and increase the air current. When cool, disconnect the

[29] Boiling for 1 min. is usually sufficient.

soda-lime tubes from the apparatus and allow to stand in the balance case until two weights taken 30 min. apart agree within 0.5 mg. The soda lime for use in this determination must be porous, not hard and unabsorptive like that sometimes used for the combustion of nitrogenous organic substances.

Carbon Dioxide by Alternative Method

19. (*a*) *Apparatus.*—The apparatus illustrated in Fig. 2, which is a modification of the usual form, may be used if desired. The principle of its operation is the same as that of the procedure described in Section 18. The apparatus consists of the following:

Tube A, for decomposition of the sample.

Tube B, to contain acid. A soda-lime tower is placed ahead of tube *B* so that air drawn through the apparatus is free of CO_2.

Micro-drier C, containing water, to act as a condenser and to retain most of the acid volatilized from tube *A*.

Bulb D, containing mossy zinc to remove the last traces of HCl.

Micro-drier E, containing H_2SO_4 to dry gases. If the sample evolves H_2S, another micro-drier, *F*, containing cadmium chloride solution is inserted between bulb *D* and micro-drier *E*.

Bulb G, containing P_2O_5 to complete the drying of the gases.

Midvale Bulb H, for absorption of CO_2. It is filled as follows: A layer of glass wool extending above the end of the outlet tube is placed in the bottom, and on this a layer of P_2O_5, about $\frac{3}{4}$ in. thick. Immediately on this is placed another layer of glass wool, and the rest of the bulb is filled with ascarite, except for a final layer of glass wool on the top.

(*b*) *Size of Sample.*—With limestone, use 0.5 g. of the sample; with burned lime, hydrated lime, etc., use 3 to 5 g. of the sample.

(*c*) *Procedure.*—The manipulations are as follows: Sweep out tubes *C*, *D*, *E*, *F*, and *G* with a current of CO_2-free air. Place in tube *B* sufficient HCl (sp. gr. 1.1) to completely decompose the sample, but so regulated in volume that the liquid level in tube *A* shall not be above the point *P*. Connect a soda-lime tower to the top of tube *B*. Introduce rapidly into tube *A* the weighed amount[5] of the prepared sample, and immediately cover it with CO_2-free water and attach to the apparatus. Then attach the weighed Midvale bulb *H*. Allow the acid from tube *B* to flow into tube *A*, the rate being governed by the rate of evolution of gas. Then gently heat tube *A* and boil to expel all CO_2[29] while drawing a slow current of air free from CO_2 through the apparatus. Continue the air current for 3 min. after removing the flame. Disconnect the Midvale bulb *H* and weigh it after it has come to equilibrium in the balance case. Use a second Midvale bulb as counterpoise in all weighings.

Sulfur Trioxide

20. Place 2 g. of the prepared sample in a small, dry beaker and stir it up with 10 ml. of cold water until all lumps are broken and the lighter particles are in suspension. Add 15 ml. of diluted HCl (1:1) and heat until solution is complete. Filter through a small paper and wash the residue thoroughly with hot water. Dilute the filtrate to 250 ml., heat to boiling, and add 10 ml. of a boiling solution of $BaCl_2$ (10 per cent) drop by drop, while stirring constantly. Stir well and allow to stand over night. Filter, wash with boiling water, ignite, and weigh as $BaSO_4$.

Total Sulfur

21. Weigh 1 g. of the prepared sample. Add approximately 0.5 g. of Na_2CO_3.

Mix thoroughly in a porcelain crucible[30] and heat gently until sintered. Then ignite for 15 min. at a temperature of approximately 1000 C., taking care to allow access of air to the contents of the crucible. Cool and place the crucible in a 250-ml. beaker and cover with hot water. Add 10 ml. of bromine water, then 30 ml. HCl (1:1) and boil until solution is complete and all bromine has been expelled. Remove the crucible, washing it with water. Add a few drops of methyl red and render the solution alkaline with NH_4OH (1:1). Boil the solution for 1 or 2 min., filter, and wash with hot water. To the filtrate add 5 ml. of HCl (1:1), adjust the volume to about 200 ml., bring the solution to boiling, and while boiling add 10 ml. of hot $BaCl_2$ (10 per cent). Allow to stand overnight. Filter, wash with hot water, ignite, and weigh as $BaSO_4$.

Phosphorus

22. Dissolve 10 g. of the prepared sample in 80 ml. diluted HNO_3 (1:1), filter, and wash the residue with hot water. Ignite, fuse the residue with a little Na_2CO_3, cool the melt, and add its nitric acid solution to the main filtrate.[31] In case the rock is rather argillaceous, mix the powder with half its weight of Na_2CO_3 and blast strongly. Dissolve the cooled melt in HNO_3 and evaporate to dryness. Take up the dry residue with diluted HNO_3 (1:2), filter, and wash the residue with hot water. Ignite the residue in platinum, and evaporate with HF and HNO_3. Finally, evaporate twice with HNO_3 alone to expel all fluorine, dissolve the residue in HNO_3 and add the solution to the main filtrate.[31] Oxidize the nitric acid solution by boiling with 10 ml. of $KMnO_4$ (1.5 per cent) until MnO_2 is precipitated. Dissolve the oxide by addition of H_2SO_3 or a sulfite free from phosphorus and boil to expel the oxides of nitrogen and sulfur. Neutralize the HNO_3 with NH_4OH and then add 1 ml. of HNO_3 (sp. gr. 1.42) for every 100 ml. of solution. Bring the solution to a temperature of 40 C., add ammonium molybdate solution[32] and shake for 10 min. Allow to stand at a temperature of not over 40 C. for 1 to 12 hr. Filter and wash ten times with KNO_3 (1 per cent). Return the precipitate to the precipitating vessel, add a measured excess of 0.1 *N* NaOH and 25 ml. of water free from CO_2 and shake or stir until the precipitate is dissolved. Dilute to 100 to 200 ml. with water free from CO_2, add 3 drops of phenolphthalein (0.2 per cent), and discharge the pink color with standard acid. Finish the titration by adding standard alkali until the reappearance of the pink color. The alkali solution should be free from carbonate and be standardized against the National Bureau of Standards' standard sample 39*e* of benzoic acid.[33] The ratio of phosphorus to NaOH should be considered as 1:23 in calculating results.[34]

Manganese

23. Dissolve 10 g. of the sample in 100 ml. of diluted HNO_3 (1:1), filter, and

[30] It is usually desirable to make a fusion in a platinum crucible. However, it has been found that with limestones very high in impurities, when the fusion is made in a muffle, the damage to platinum ware is considerable. It has been proved that the use of porcelain crucibles introduces no appreciable error.

[31] In limestones very low in phosphorus it may be deemed desirable to concentrate the phosphorus from a considerable weight of sample. In this case, proceed as directed by F. Hinden (*Zeitschr. Anal. Chemie*, Vol. 54 p. 214 (1915)), as follows: To the nitric acid solution of the large sample add a few drops of $FeCl_3$ solution and then NH_4OH until the liquid becomes slightly turbid. Then add 0.5 g. of pure precipitated $CaCO_3$ and boil for 5 min. Filter, dissolve the precipitate in diluted HNO_3, and proceed as above.

[32] See Blair, "The Chemical Analysis of Iron," 8th Edition, p. 92.

[33] See *Journal*, Am. Chem. Soc., Vol. 34, p. 1027 (1912), and Vol. 35, p. 1309 (1913).

[34] Titanium and vanadium, which interfere in this determination, should not be encountered in appreciable amounts in this class of material.

Instead of the specified alkalimetric titration method, the phosphorus in the phosphomolybdate may be determined as magnesium pyrophosphate (U. S. Geological Survey *Bulletin 700*, pp. 178-179), or by the reductor method (A. A. Blair, "Chemical Analysis of Iron," 8th Edition, pp. 91-92).

wash the residue with hot water. Ignite the residue in platinum, fuse with a little Na_2CO_3, and add the nitric acid solution of the melt to the main filtrate. Dilute the solution to 150 ml. and add 0.5 g. of sodium bismuthate.[35] Heat for a few minutes, or until the pink color has disappeared and MnO_2 has precipitated. If MnO_2 does not precipitate add more bismuthate. Clear the solution by adding a few drops of a saturated solution of sodium bisulfite or other suitable reducing agent free from chlorides, and boil to expel all oxides of nitrogen and sulfur. Cool to 15 C., add an excess of sodium bismuthate, agitate, and let stand for a few minutes. Add 50 ml. of HNO_3 (3 per cent) and filter through asbestos. Wash with HNO_3 (3 per cent) until the washings run through colorless. Add a measured excess of a standard $FeSO_4$ solution and titrate back with a standard $KMnO_4$ solution, the strength of which has been determined by means of the National Bureau of Standards' standard sample 40c of sodium oxalate.[36]

Ferrous Iron in the Absence of Carbonaceous Matter[37]

24. Introduce the powder, one to several grams, into a stout flask of about 200 to 250-ml. capacity and boil with a little water until all air is expelled. While still boiling, add diluted H_2SO_4 a little at a time until effervescence ceases, then a further amount. Calcium sulfate precipitates, but the iron will remain in solution. Remove the flame and tightly insert a stopper through which passes a small stopcock funnel. When the flask is cool or nearly so, pour cold water into the funnel and cautiously open the cock so the water may be drawn into the flask, pouring more water into the funnel as fast as it empties until the solution amounts to 100 to 150 ml. Such precaution to exclude air is hardly necessary in most cases, however, since in the presence of H_2SO_4, the oxidation of ferrous iron is exceedingly slow. It is ordinarily quite sufficient to equalize the internal and external pressures by opening the cock, removing the stopper, and pouring in cold water. Bring the flask under a burette containing 0.05 *N* $KMnO_4$ solution and titrate the iron without delay. With a carbonate which is wholly decomposable without the aid of heat, solution may be accomplished in a flask filled with CO_2, using cold acid, whereby the danger of attack of silicates is lessened if these are present. If the preference is for the $K_2Cr_2O_7$ method of titration, HCl may be used instead of H_2SO_4, in absence of MnO_2. In this case there is, of course, no separation of an insoluble calcium salt, a fact which renders easier the subsequent determination of the iron in any insoluble residue the limestone may yield. The ferrous iron thus found is mostly, if not altogether, that existing as carbonate.[38]

Ferrous Iron In Presence of Carbonaceous Matter[37]

25. Decompose the powder in a flask with diluted H_2SO_4 in an atmosphere of CO_2. With limestones and active agitation no heat need be used, but with dolomites it will be necessary. Quickly filter the solution through asbestos (in

[35] The persulfate method may be employed, if desired.

[36] The separation of manganese with the R_2O_3 precipitate by the use of bromine is not recommended on account of the incompleteness of the precipitation. Separation of manganese by means of ammonium sulfide after the ammonia precipitation is also undesirable on account of the introduction of the reagent and the slimy character of the precipitate.

See also footnote 25 concerning the contamination of magnesium pyrophosphate by manganese pyrophosphate and the need of corrective measures when manganese is present in the material under analysis.

[37] In limestones, the presence of carbonaceous matter renders the exact or even approximate determination of ferrous iron often impossible. Nevertheless, even in its presence acceptable results are sometimes obtainable if the amount of such matter is small and if it does not give with acid a colored solution. Occasionally limestones show films of manganese peroxide which likewise interfere with the determination.

[38] If a determination of ferrous iron in the insoluble residue is desired, see U. S. Geological Survey *Bulletin 700*, p. 265.

an atmosphere of CO_2 if much iron is present), wash the residue and filter a few times with water, and titrate the filtrate at once with 0.05 *N* $KMnO_4$. If it is colored by organic matter, the result may be in error. As above, HCl and titration by $K_2Cr_2O_7$ may be used in absence of MnO_2. In either case it is important to allow the acid to act no longer than is necessary and to filter quickly. A determination of the ferrous iron in the insoluble matter is usually not worth attempting, because of the admixed organic matter.

Available Lime Index

26. (*a*) *Available Lime Index.*—"The available lime index" of high calcium quicklime and hydrated lime designates those constituents which enter into the reaction under the conditions of this specified method. The interpretation of results obtained by the following method shall be restricted by this definition.

(*b*) *Standard Hydrochloric Acid Solution.*—Prepare a solution containing 13.7 ml. of HCl (sp. gr. 1.18) per liter. This solution will be slightly stronger than necessary. Standardize the HCl solution[39] against 0.85 g. of pure, dry Na_2CO_3 using methyl orange as the indicator. Adjust the solution either by the addition of water if too strong, or by the addition of HCl if too weak, so that 0.85 g. of Na_2CO_3 exactly neutralizes 90 ml. of the standard HCl solution. One milliliter of the standard HCl solution is equivalent to 1 per cent CaO.

(*c*) *Rapid Sugar Method.*—Weigh 0.5 g. of the prepared sample (passing a No. 100 (149-micron) sieve) and brush carefully into a 300-ml. Erlenmeyer flask containing about 20 ml. of distilled water (Note 1), and immediately stopper the flask loosely with a rubber stopper. Disperse the sample thoroughly in the water by a swirling motion, heat to boiling, and boil for 2 min. Remove the stopper, add 150 ml. of distilled water at room temperature, and then add 15 g. of granulated sugar (sucrose) (Note 2). Stopper the flask, shake vigorously at intervals for a 5-min. period, and allow to stand for 30 min., but not longer than 1 hr. Add 2 drops of phenolphthalein indicator, wash down the stopper and sides of the flask with distilled water, and titrate (in the original flask) with the standard HCl solution (Note 3).

NOTE 1.—It is particularly important to have a little water in the flask before adding the sample. This is especially true in the case of quicklime, since by adding water on top of a dry charge in a flask there is frequently a tendency for the material to cake and form lumps difficult to completely dissolve in the sugar solution later. On the other hand, if the lime is added to a little water, conditions are favorable to a thorough dispersion of fine particles to produce a more rapid solution. In the case of quicklime, probably some slaking action occurs to facilitate the dispersion and solution.

NOTE 2.—The 15 g. of sugar and the 150 ml. of water used are only approximate amounts. It is not necessary to weigh the sugar each time. A small measure can be employed, such as a spoon which when level full holds slightly more than 15 g. This saves time in running a large number of samples, and just as accurate results are obtained as when the sugar is carefully weighed. A little excess weight of sugar always insures at least a 10 per cent sugar solution.

NOTE 3.—In titrating, first add without shaking about 90 per cent of the acid requirement from a 100-ml. burette. Then shake the flask and finish the titration more carefully, but as rapidly as possible (shaking the flask vigorously), to the first complete disappearance of pink color. Note the reading in per cent and ignore return of color. Unless the operator is familiar with previous analyses of the lime under test, and in cases where the available lime content varies to extremes, it is good practice to run a preliminary test by slow titration to determine the proper amount to add without first shaking the flask. Inasmuch as the method is very simple, this entails no great hardship.

[39] This solution may also be standardized against National Bureau of Standards' standard sample 39 of benzoic acid or standard sample 84 of potassium acid phthalate through an NaOH solution.

APPENDIX

ANALYSIS OF LIMESTONE OR LIME PRODUCTS

(Name of Company)

Date..... Lab. No.....

Name..... Date Rec'd.....

Material.....

Sample Marked.....

CONSTITUENTS DETERMINED			CONSTITUENTS CALCULATED[a]		
Name	Formula	Per cent	Name	Formula	Per cent
Silicon dioxide	SiO_2		Calcium carbonate	$CaCO_3$	
Iron oxide	Fe_2O_3		Calcium hydroxide	$Ca(OH)_2$	
Aluminum oxide	Al_2O_3		Magnesium carbonate	$MgCO_3$	
Calcium oxide	CaO		Magnesium hydroxide	$Mg(OH)_2$	
Magnesium oxide	MgO		Calcium sulfate	$CaSO_4$	
Total sulfur	S				
Sulfur trioxide	SO_3				
Phosphorus pentoxide	P_2O_5				
Carbon dioxide	CO_2				
Water at 120 C	H_2O				
Water total	H_2O				
Insoluble matter					
Loss on ignition					
Available lime index					
Total neutralizing value in terms of $CaCO_3$ Calculated					
Total neutralizing value in terms of $CaCO_3$ Determined					
Plasticity					
Soundness					

[a] Method for calculation *must* be noted for each constituent.

SIEVE ANALYSIS

Sieve No.						
Opening in microns						
Percentage passing						

REMARKS

NOTE.—Unless otherwise noted all determinations have been made according to methods prescribed by the American Society for Testing Materials.

Signed.....

Standard Definitions of

TERMS RELATING TO LIME[1]

A.S.T.M. Designation: C 51 - 44

ADOPTED, 1928; REVISED, 1939, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 51; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Lime.—A general term which includes the various chemical and physical forms of quicklime, hydrated lime, and hydraulic lime used for any purpose.

Quicklime.—A calcined material, the major part of which is calcium oxide or calcium oxide in natural association with a lesser amount of magnesium oxide, capable of slaking with water.

Lump Lime.—Quicklime as it comes from the kilns.

Lump Lime Screened.—Lump lime after forking or screening to remove the finer portion.

NOTE.—The portion removed is usually that which will pass a ½-in. sieve.

Pulverized Lime.—Quicklime which will pass a fine sieve of specified size.

NOTE.—The size of the sieve is usually ¼-in.

Hydrated Lime.—A dry powder obtained by treating quicklime with water enough to satisfy its chemical affinity for water under the conditions of its hydration.

NOTE.—It consists essentially of calcium hydroxide or a mixture of calcium hydroxide and magnesium oxide and magnesium hydroxide.

Liming Material.—A general term which includes all of the various chemical and physical forms of lime, limestone, mollusk shells, and marl whose calcium and magnesium content is capable of neutralizing soil acidity.

Air-Slaked Lime.—The product containing various proportions of the oxides, hydroxides, and carbonates of calcium and magnesium which results from the excessive exposure of quicklime to the air.

Calcia.—The chemical compound CaO, calcium oxide.

Magnesia.—The chemical compound MgO, magnesium oxide.

Building or Construction Lime.—A lime whose chemical and physical characteristics and method of processing make it suitable for the ordinary or special construction uses of the product.

Chemical Lime.—A lime whose chemical and physical characteristics and method of processing make it suitable for one or more of the many and varied chemical and industrial uses of the product.

Agricultural Lime.—A lime whose calcium and magnesium content is capable of neutralizing soil acidity.

Available Lime.—Those constituents of a lime which enter into a desired reaction under the conditions of a specific method or process.

[1] Under the standardization procedure of the Society, these definitions are under the jurisdiction of the A.S.T.M. Committee C-7 on Lime.

[2] Prior to adoption as standard, these definitions were published as tentative from 1922 to 1928, being revised in 1924 and 1928.

A "tentative revision" of this standard appears in the Tentative Revisions of Standards section at the back of this book.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: A49.1-1941

Standard Specifications for

GYPSUM[1]

A.S.T.M. Designation: C 22 - 41

ADOPTED, 1925; REVISED, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 22; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. (*a*) These specifications cover gypsum, calcium sulfate combined with two molecules of water in crystalline form and having the approximate chemical formula $CaSO_4 \cdot 2H_2O$.

(*b*) No material may be considered gypsum within the meaning of these specifications that contains less than 64.5 per cent by weight of $CaSO_4 \cdot 2H_2O$.

Size

2. Gypsum may be crushed and ground to the size specified by the purchaser.

Chemical Composition

3. The chemical composition, within the limits prescribed by Section 1, shall be a matter of contract.

Sampling

4. (*a*) *Bulk.*—When gypsum is shipped in bulk, samples of about 5 lb. each shall be taken at regular intervals during the complete loading or unloading of the carrier, in such a way as to accumulate at least 200 lb. of material and to represent correctly the percentages of both the coarse particles and the fines in the shipment. This shall be broken to pass a 1-in. round-hole screen, thoroughly mixed, and reduced by quartering to provide not less than a 1-lb. sample for the laboratory.

(*b*) *Packages.*—When gypsum is shipped in packages, at least 3 per cent of the packages shall be sampled. Samples shall be taken from both the surface and the center of the packages. These samples shall then be broken, mixed, and quartered as directed above.

Laboratory Samples

5. Each laboratory sample shall be placed immediately in an airtight container and shipped to the laboratory for test.

Methods of Testing

6. The chemical analysis and physical properties of gypsum shall be determined in accordance with the Standard Methods of Testing Gypsum and Gypsum Products (A.S.T.M. Designation: C 26)

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-11 on Gypsum.

[2] Prior to adoption as standard, these specifications were published as tentative from 1919 to 1925, being revised in 1920, 1921, 1923, 1924, and 1925.

of the American Society for Testing Materials.[3]

Packing and Marking

7. (*a*) Gypsum may be shipped either in packages or in bulk.

(*b*) When shipped in packages for resale, the following information (Note) shall be legibly marked on each package or on a tag of suitable size attached thereto:

Name of manufacturer,
Description of material, and
Net and gross weights of package.

(*c*) When shipped in bulk, a card containing the required information shall be conspicuously placed in the carrier.

NOTE.—State laws may require additional information.

Inspection

8. Inspection may be made either at the point of shipment or at the point of delivery. The inspector representing the purchaser shall have free access to the carriers being loaded for shipment to the purchaser. He shall be afforded all reasonable facilities for inspection and sampling, which shall be so conducted as not to interfere unnecessarily with the loading of the carriers.

Rejection

9. Any rejection shall be based upon the specific cause of failure to conform to the requirements of these specifications, and shall be reported to the seller within 10 working days from the receipt of the shipment by the purchaser.

Rehearing

10. Claims for rehearing shall be valid only if made within 20 working days from receipt of notice of specific cause for rejection.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: A49.3-1940

Standard Specifications for

GYPSUM PLASTERS[1]

A.S.T.M. Designation: C 28 - 40

ADOPTED, 1921; REVISED, 1927, 1930, 1939, 1940.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 28; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover four gypsum plasters: namely,
Gypsum ready-sanded plaster,
Gypsum neat plaster,
Gypsum wood-fibered plaster, and
Calcined gypsum for finishing coat.

Calcined Gypsum

2. The term calcined gypsum in these specifications refers to $CaSO_4 \cdot \frac{1}{2}H_2O$, calculated from the SO_3 content.

CHEMICAL AND PHYSICAL PROPERTIES

Gypsum Ready-Sanded Plaster

Description

3. Gypsum ready-sanded plaster is a plastering material in which the predominating cementitious material is calcined gypsum, and which is mixed at the mill with all the constituent parts, including sand, in their proper proportion. Only the addition of water is required to make it ready for use.

Composition

4. The following shall govern the composition of gypsum ready-sanded plaster for the desired coats:

(*a*) *Scratch or First Coat Plaster.*—Gypsum ready-sanded plaster used for scratch or first coat shall contain not more than two-thirds by weight of sand. The other one-third shall contain not less than 60.5 per cent by weight of calcined gypsum. The remainder may consist of materials to control the working quality, the setting time, and the fibering.

(*b*) *Brown or Second Coat Plaster.*—Gypsum ready-sanded plaster used for browning or second coat shall contain not more than three-fourths by weight of sand. The other one-fourth shall contain not less than 60.5 per cent by weight of calcined gypsum. The remainder may consist of materials to control the working quality, the setting time, and the fibering.

Time of Setting

5. Gypsum ready-sanded plaster shall conform to the following requirements as to time of setting:

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-11 on Gypsum.

[2] Prior to adoption as standard, these specifications were published as tentative from 1920 to 1921, being revised in 1921.

(*a*) *Scratch or First Coat Plaster.*—Not less than 1½ nor more than 7 hr.

(*b*) *Brown or Second Coat Plaster.*—Not less than 2 nor more than 6 hr.

Tensile Strength

6. Gypsum ready-sanded plaster shall conform to the following requirements as to tensile strength:

(*a*) *Scratch or First Coat Plaster.*—Not less than 125 psi.

(*b*) *Brown or Second Coat Plaster.*—Not less than 75 psi.

Gypsum Neat Plaster

Description

7. Gypsum neat plaster is a plastering material in which not less than 60.5 per cent of the cementitious material is calcined gypsum, and which is mixed at the mill with other materials.

Composition

8. Gypsum neat plaster shall contain not less than 60.5 per cent by weight of calcined gypsum. The remainder may consist of materials to control the working quality, the setting time, and the fibering.

Time of Setting

9. Gypsum neat plaster when mixed with three parts by weight of standard sand shall set in not less than 2 nor more than 32 hr.

Tensile Strength

10. Gypsum neat plaster as tested with two parts of standard sand shall have a tensile strength of not less than 125 psi.

Gypsum Wood-Fibered Plaster

Description

11. Gypsum wood-fibered plaster is a gypsum plaster in which wood fiber is used as an aggregate.

Composition

12. Gypsum wood-fibered plaster shall contain not less than 60.5 per cent by weight of calcined gypsum and not less than 1 per cent by weight of wood fiber made from a nonstaining wood. The remainder may consist of materials to control the working quality and setting time.

Time of Setting

13. Gypsum wood-fibered plaster shall set in not less than 1½ nor more than 8 hr.

Tensile Strength

14. Gypsum wood-fibered plaster shall have a tensile strength of not less than 125 psi.

Calcined Gypsum for Finishing Coat

Description

15. Calcined gypsum for finishing coat may or may not contain retarder, and may be classified in two grades: namely, "white" and "gray."

Fineness

16. Calcined gypsum for finishing coat shall all pass a No. 14 (1410-micron) sieve, and not less than 60 per cent shall pass a No. 100 (149-micron) sieve.[3]

Time of Setting

17. Calcined gypsum for finishing coat, when not retarded, shall set in not less than 20 nor more than 40 min., and when retarded, shall set in not less than 40 min.

Tensile Strength

18. Calcined gypsum for finishing coat shall have a tensile strength of not less than 200 psi.

[3] Detailed requirements for these sieves are given in the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

SAMPLING

Sampling

19. At least 3 per cent of the packages shall be sampled, and shall be so selected as to be representative of the shipment. Samples shall be taken from both the surface and the center of each package so selected. The material so obtained shall be thoroughly mixed and reduced by quartering to provide not less than a 15-lb. sample for the laboratory.

Laboratory Samples

20. Each laboratory sample shall be placed immediately in an airtight container and shipped to the laboratory for test.

METHODS OF TESTING

Methods of Testing

21. The chemical analysis and physical properties of gypsum plasters shall be determined in accordance with the Standard Methods of Testing Gypsum and Gypsum Products (A.S.T.M. Designation: C 26) of the American Society for Testing Materials.[4]

NOTE.—Distilled water shall be used for conducting all tests.

PACKING AND MARKING

Packing and Marking

22. (*a*) Gypsum plasters shall be dry and free from lumps, and shall be shipped in packages.

(*b*) When shipped for resale, the following information shall be legibly marked on each package or on a tag of suitable size attached thereto:

Name of manufacturer,
Brand,
Description, and
Net and gross weights of package.

INSPECTION AND REJECTION

Inspection

23. Inspection may be made either at the point of shipment or at the point of delivery. The inspector representing the purchaser shall have free access to the carrier being loaded for shipment to the purchaser. He shall be afforded all reasonable facilities for inspection and sampling, which shall be so conducted as not to interfere unnecessarily with the loading of the carriers.

Rejection

24. Any rejection shall be based upon the specific cause of failure to conform to the requirements of these specifications, and shall be reported to the seller within 10 working days from the receipt of the shipment by the purchaser.

Rehearing

25. Claim for rehearing shall be valid only if made within 20 working days from receipt of notice of specific cause for rejection.

[4] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: A49.4-1940

Standard Specifications for

GYPSUM MOLDING PLASTER[1]

A.S.T.M. Designation: C 59 - 40

ADOPTED, 1929; REVISED, 1930, 1940.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 59; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover gypsum molding plaster, a material consisting essentially of calcined gypsum for use in making interior embellishments, cornices, etc.

Composition

2. Gypsum molding plaster shall contain not less than 80 per cent of calcined gypsum ($CaSO_4 \cdot \frac{1}{2}H_2O$).

Time of Setting

3. Gypsum molding plaster shall set in not less than 20 nor more than 40 min.

Tensile Strength

4. Gypsum molding plaster shall have a tensile strength of not less than 200 psi.

Fineness

5. Gypsum molding plaster shall all pass a No. 30 (590-micron) sieve and not less than 90 per cent shall pass a No. 100 (149-micron) sieve.[3]

Sampling

6. At least 3 per cent of the packages shall be sampled and shall be so selected as to be representative of the shipment. Samples shall be taken from both the surface and the center of each packag so selected. The material so obtained shall be thoroughly mixed and reduced by quartering to provide not less than a 15-lb. sample for the laboratory.

Laboratory Samples

7. Each laboratory sample shall be placed immediately in an airtight container and shipped to the laboratory for test.

Methods of Testing

8. The chemical analysis and physical properties of gypsum molding plaster shall be determined in accordance with the Standard Methods of Testing Gypsum and Gypsum Products (A.S.T.M. Designation: C 26) of the American Society for Testing Materials.[4]

NOTE.—Distilled water shall be used for conducting all tests.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-11 on Gypsum.

[2] Prior to adoption as standard, these specifications were published as tentative from 1926 to 1929, being revised in 1929.

[3] Detailed requirements for these sieves are given in the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

[4] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Packing and Marking

9. (*a*) Gypsum molding plaster shall be dry and free from lumps and shall be shipped in packages.

(*b*) When shipped for resale, the following information shall be legibly marked on each package or on a tag of suitable size attached thereto:

Name of manufacturer,
Brand,
Description, and
Net and gross weights of package.

Inspection

10. Inspection may be made either at the point of shipment or at the point of delivery. The inspector representing the purchaser shall have free access to the carrier being loaded for shipment to the purchaser. He shall be afforded all reasonable facilities for inspection and sampling, which shall be so conducted as not to interfere unnecessarily with the loading of the carriers.

Rejection

11. Any rejection shall be based upon the specific cause of failure to conform to the requirements of these specifications, and shall be reported to the seller within 10 working days from the receipt of the shipment by the purchaser.

Rehearing

12. Claim for rehearing shall be valid only if made within 20 working days from receipt of notice of specific cause for rejection.

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ASA NO.: A49.5-1940

Standard Specifications for

GYPSUM POTTERY PLASTER[1]

A.S.T.M. Designation: C 60 - 40

ADOPTED, 1929; REVISED, 1930, 1940.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 60; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover gypsum pottery plaster, a material consisting essentially of calcined gypsum for use in making pottery molds.

Composition

2. Gypsum pottery plaster shall contain not less than 90 per cent of calcined gypsum ($CaSO_4 \cdot \frac{1}{2}H_2O$).

Time of Setting

3. Gypsum pottery plaster shall set in not less than 20 nor more than 40 min.

Tensile Strength

4. Gypsum pottery plaster shall have a tensile strength of not less than 250 psi.

Fineness

5. Gypsum pottery plaster shall all pass a No. 30 (590-micron) sieve and not less than 94 per cent shall pass a No. 100 (149-micron) sieve.[3]

Sampling

6. At least 3 per cent of the packages shall be sampled and shall be so selected as to be representative of the shipment. Samples shall be taken from both the surface and the center of each package so selected. The material so obtained shall be thoroughly mixed and reduced by quartering to provide not less than a 15-lb. sample for the laboratory.

Laboratory Samples

7. Each laboratory sample shall be placed immediately in an airtight container and shipped to the laboratory for test.

Methods of Testing

8. The chemical analysis and physical properties of gypsum pottery plaster shall be determined in accordance with the Standard Methods of Testing Gypsum and Gypsum Products (A.S.T.M. Designation: C 26) of the American Society for Testing Materials,[4] except

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-11 on Gypsum.

[2] Prior to adoption as standard, these specifications were published as tentative from 1926 to 1929, being revised in 1929.

[3] Detailed requirements for these sieves are given in the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

[4] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

that in the measurement of the time of setting and tensile strength the plaster and water shall be mixed in the proportion of 100 g. of plaster to 60 ml. of water.

NOTE.—Distilled water shall be used for conducting all tests.

Packing and Marking

9. (*a*) Gypsum pottery plaster shall be dry and free from lumps and shall be shipped in packages.

(*b*) When shipped for resale, the following information shall be legibly marked on each package or on a tag of suitable size attached thereto:

Name of manufacturer,
Brand,
Description, and
Net and gross weights of package.

Inspection

10. Inspection may be made either at the point of shipment or at the point of delivery. The inspector representing the purchaser shall have free access to the carrier being loaded for shipment to the purchaser. He shall be afforded all reasonable facilities for inspection and sampling, which shall be so conducted as not to interfere unnecessarily with the loading of the carriers.

Rejection

11. Any rejection shall be based upon the specific cause of failure to conform to the requirements of these specifications, and shall be reported to the seller within 10 working days from the receipt of the shipment by the purchaser.

Rehearing

12. Claim for rehearing shall be valid only if made within 20 working days from receipt of notice of specific cause for rejection.

APPROVED AS
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BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: A66.1-1941

Standard Specifications for

KEENE'S CEMENT[1]

A.S.T.M. Designation: C 61 - 40

ADOPTED, 1930; REVISED, 1940.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 61; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover Keene's cement, anhydrous calcined gypsum the set of which is accelerated by the addition of other materials.

NOTE.—Various grades of Keene's cement of different fineness and setting time are available. These specifications are directly applicable to the grades manufactured for use in the base and finish coats of plastering. Grades intended for use in Scagliola, castings, and other special purposes should conform to the requirements of these specifications in all respects except those for fineness and time of set, which will vary according to the particular use for which the grade is designed.

Time of Setting

2. Keene's cement shall set in not less than 1 nor more than 4 hr.

Tensile Strength

3. The cement shall have a tensile strength of not less than 400 psi.

Fineness

4. The cement shall all pass a No. 14 (1410-micron) sieve, not less than 98 per cent shall pass a No. 40 (420-micron) sieve, and not less than 80 per cent shall pass a No. 100 (149-micron) sieve.[3]

Combined Water

5. The cement shall not show a combined water content of more than 2 per cent.

Sampling

6. At least 3 per cent of the packages shall be sampled and shall be so selected as to be representative of the shipment. Samples shall be taken from both the surface and the center of each package so selected. The material so obtained shall be thoroughly mixed and reduced by quartering to provide not less than a 15-lb. sample for the laboratory.

Laboratory Samples

7. Each laboratory sample shall be placed immediately in an airtight container and shipped to the laboratory for test.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-11 on Gypsum.

[2] Prior to adoption as standard, these specifications were published as tentative from 1926 to 1930, being revised in 1928, 1929, and 1930.

[3] Detailed requirements for these sieves are given in the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Methods of Testing

8. The chemical analysis and physical properties of Keene's cement shall be determined in accordance with the Standard Methods of Testing Gypsum and Gypsum Products (A.S.T.M. Designation: C 26) of the American Society for Testing Materials,[4] except that in the determination of the time of setting and tensile strength, the consistency used shall be such that a 150-g. modified Vicat needle, described in Section 15 of the Standard Methods C 26, shall give a penetration of 30 ± 2 mm., 20 sec. after release, the measurement being made 5 min. after the addition of the Keene's cement to the gaging water.

NOTE.—A 20-min. mixing and soaking period for the Keene's cement and water with occasional stirring to remove entrained air or gas bubbles is required before filling the molds in the strength determination. The use of 0.1 per cent retarder in determining the testing consistency is necessary when working with quick-setting cements.

Packing and Marking

9. (*a*) Keene's cement shall be dry and free from lumps and shall be shipped in packages.

(*b*) When shipped for resale, the following information shall be legibly marked on each package or on a tag of suitable size attached thereto:

Name of manufacturer,
Brand,
Description, and
Net and gross weights of package.

Inspection

10. Inspection may be made either at the point of shipment or at the point of delivery. The inspector representing the purchaser shall have free access to the carriers being loaded for shipment to the purchaser. He shall be afforded all reasonable facilities for inspection and sampling, which shall be so conducted as not to interfere unnecessarily with the loading of the carriers.

Rejection

11. Any rejection shall be based upon the specific cause of failure to conform to the requirements of these specifications and shall be reported to the seller within 10 working days from the receipt of the shipment by the purchaser.

Rehearing

12. Claims for rehearing shall be valid only if made within 20 working days from receipt of notice of specific cause for rejection.

[4] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

APPENDIX

FIELD TEST FOR KEENE'S CEMENT

In many instances it is desirable to have a simple test whereby Keene's cement may be identified. Such a test will not indicate the quality of the cement, which should be determined by the laboratory tests enumerated in the body of these specifications, but is of especial value to plasterers, material dealers, and superintendents.

The following procedure is satisfactory for this purpose:

Take a cupful of the material, mix with water to the consistency of a thick paste, and pour upon a plate or piece of glass. Let stand until fairly firm and definite signs of set having begun are manifest. This will be some time less than 2 hr., depending on climatic conditions.

Divide the sample, allowing one half to remain undisturbed. Take the other half and break it down adding a little water, remix, and then allow it to "set up" again on the plate or glass.

If the material is Keene's cement, the remixed portion will, within a few hours, become quite as hard and strong as the portion that was not remixed.

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Standard Specifications for
CALCINED GYPSUM FOR DENTAL PLASTERS[1]

A.S.T.M. Designation: C 72 – 40

ADOPTED, 1930; REVISED, 1940.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 72; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover calcined gypsums having specific properties with reference to time of setting, fineness, and water-carrying capacity, for use in the preparation of dental plasters. Three grades are covered, as follows:

Grade Q (Quick setting),
Grade M (Medium setting), and
Grade S (Slow setting).

Composition

2. Calcined gypsum for use in the preparation of dental plasters shall contain not less than 93 per cent of calcined gypsum ($CaSO_4 \cdot \frac{1}{2}H_2O$).

Time of Setting

3. The calcined gypsum shall conform to the following requirements as to time of setting:

Grade Q.—Not less than 2 nor more than 4 min.

Grade M.—Not less than 6 nor more than 12 min.

Grade S.—Not less than 20 nor more than 40 min.

Tensile Strength

4. The calcined gypsum shall have a tensile strength of not less than 300 psi.

Fineness

5. The calcined gypsum shall all pass a No. 30 sieve (590-micron) and not less than 95 per cent shall pass a No. 100 (149-micron) sieve.[3]

Sampling

6. At least 3 per cent of the packages shall be sampled and shall be so selected as to be representative of the shipment. Samples shall be taken from both the surface and the center of each package so selected. The material so obtained shall be thoroughly mixed and reduced by quartering to provide not less than a 15-lb. sample for the laboratory.

Laboratory Samples

7. Each laboratory sample shall be placed immediately in an airtight con-

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-11 on Gypsum.

[2] Prior to adoption as standard, these specifications were published as tentative from 1928 to 1930, being revised in 1929 and 1930.

[3] Detailed requirements for these sieves are given in the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

A "tentative revision" of this standard appears in the Tentative Revisions of Standards section at the back of this book.

tainer and shipped to the laboratory for test.

Methods of Testing

8. The chemical analysis and physical properties of calcined gypsum for use in the preparation of dental plasters shall be determined in accordance with the Standard Methods of Testing Gypsum and Gypsum Products (A.S.T.M. Designation: C 26) of the American Society for Testing Materials,[4] except that in the determination of the time of setting and the tensile strength the calcined gypsum and water shall be mixed in the proportion of 100 g. of calcined gypsum to 60 ml. of water. The calcined gypsum shall be allowed to soak in distilled water at 70 F. (21 C.) for 30 sec., and shall then be stirred for 30 sec. and placed immediately in the molds.

Packing and Marking

9. (*a*) Calcined gypsum for use in the preparation of dental plasters shall be dry and free from lumps and shall be shipped in packages.

(*b*) When shipped for resale, the following information shall be legibly marked on each package or on a tag of suitable size attached thereto:

Name of manufacturer,
Brand,
Description, and
Net and gross weights of package.

Inspection

10. Inspection may be made either at the point of shipment or at the point of delivery. The inspector representing the purchaser shall have free access to the carrier being loaded for shipment to the purchaser. He shall be afforded all reasonable facilities for inspection and sampling, which shall be so conducted as not to interfere unnecessarily with the loading of the carriers.

Rejection

11 Any rejection shall be based upon the specific cause of failure to conform to the requirements of these specifications and shall be reported to the seller within 10 working days from the receipt of the shipment by the purchaser.

Rehearing

12. Claim for rehearing shall be valid only if made within 20 working days from receipt of notice of specific cause for rejection.

[4] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

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ASA NO.: A69.1-1942

Standard Specifications for

GYPSUM WALL BOARD[1]

A.S.T.M. Designation: C 36 - 42

ADOPTED, 1934; REVISED, 1942.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 36; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover gypsum wall board, which is designed to be used without the addition of plaster for walls, ceilings, or partitions and affords a surface suitable to receive decoration.

Composition

2. Gypsum wall board shall consist of an incombustible core of gypsum with or without fiber, but not exceeding 15 per cent of fiber by weight, and surfaced with paper or other fibrous material firmly bonded to the core.

Flexural Strength

3. When tested, either face up or face down, in accordance with the Standard Methods of Testing Gypsum and Gypsum Products (A.S.T.M. Designation: C 26) of the American Society for Testing Materials,[3] specimens taken from the gypsum wall board shall carry not less than the following loads without showing a breaking of the bond between the surfacing and the core:

Thickness, in.	Load, lb. Bearing Edges Across Fiber of Surfacing	Load, lb. Bearing Edges Parallel to Fiber of Surfacing
$\frac{1}{4}$	50	24
$\frac{3}{8}$	80	34
$\frac{1}{2}$	105	45

Sampling

4. At least 1 per cent of the number of gypsum wall boards in a shipment, but not less than ten boards, shall be so selected as to be representative of the shipment. Twenty-five per cent of such selected boards, but not less than ten individual boards, shall constitute a sample for purpose of tests. When a shipment consists of more than one car or carrier load, a sample shall be taken from each car or carrier.

Dimensions, Weights, and Permissible Variations

5. (*a*) *Thickness.*—The nominal thickness of gypsum wall board shall be $\frac{1}{4}$ in., $\frac{3}{8}$ in., or $\frac{1}{2}$ in., with permissible variations in the nominal thickness of plus or minus

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-11 on Gypsum.

[2] Prior to their present adoption as standard, these specifications were published as tentative from 1921 to 1925, being revised in 1922, 1924, and 1925. They were adopted in 1925 but withdrawn and replaced in 1933 by C 36 - 33 T which were published as tentative from 1931 to 1934.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

$\frac{1}{64}$ in., and with permissible local variations of plus or minus $\frac{1}{32}$ in. from the nominal thickness. Edges, or edges and ends of $\frac{3}{8}$ in. and $\frac{1}{2}$ in. board may be plain or recessed to receive a joint reinforcing strip; if recessed, the depression shall be not less than 0.02 in.

(*b*) *Width.*—The nominal width of gypsum wall board shall be 24 in., 32 in., 36 in., or 48 in., with a permissible variation of $\frac{3}{32}$ in. under the specified width.

(*c*) *Length.*—The nominal length of $\frac{1}{4}$-in. gypsum wall board shall be from 4 ft. to 12 ft. and of $\frac{3}{8}$-in. and $\frac{1}{2}$-in. board from 4 ft. to 14 ft., inclusive, with permissible variations of plus or minus $\frac{1}{4}$ in. from the specified length.

(*d*) *Weight.*—The weight of gypsum wall board per 1000 sq. ft. shall conform to the following requirements:

Thickness, in.	Weight per 1000 sq. ft., lb. Minimum	Maximum
$\frac{1}{4}$	900	1500
$\frac{3}{8}$	1350	2000
$\frac{1}{2}$	1800	3000

Finish

6. The surfaces of gypsum wall board shall be true and free from imperfections that would render the wall board unfit for use with or without decoration. The edges and ends shall be straight and solid. The corners shall be square with a permissible variation of $\frac{1}{8}$ in. in the full width of the board.

NOTE 1.—Gypsum wall board $\frac{1}{2}$-in. in thickness is available either with square edges or V-tongue and grooved edges. Recessed board is available only with square edges.

NOTE 2.—Gypsum wall board is also available with one surface covered with aluminum or other heat-reflecting type of foil.

Packing and Marking

7. (*a*) Gypsum wall board shall be shipped so as to be kept dry and free from moisture.

(*b*) When shipped for resale, the name of the manufacturer and the brand shall be legibly marked on each board or package.

Inspection

8. Inspection may be made either at the point of shipment or at the point of delivery. The inspector representing the purchaser shall have free access to the carriers being loaded for shipment to the purchaser. He shall be afforded all reasonable facilities for inspection and sampling, which shall be so conducted as not to interfere unnecessarily with the loading of the carriers.

Rejection

9. Any rejection shall be based upon failure to conform to the requirements of these specifications and shall be reported to the seller within 10 working days from the receipt of the shipment by the purchaser. The notice of rejection shall contain a specific statement of the respects in which the boards have failed to conform to the requirements of these specifications.

Rehearing

10. Claims for rehearing shall be valid only if made within 20 working days from the receipt of notice of specific cause for rejection.

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ASA NO.: A67.1-1942

Standard Specifications for

GYPSUM LATH[1]

A.S.T.M. Designation: C 37 – 42

ADOPTED, 1934; REVISED, 1940, 1942.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 37; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover plain, perforated, and other mechanical key types of gypsum lath, which are designed to be used as a base for the reception of gypsum plaster.

Composition

2. Gypsum lath shall consist of an incombustible core of gypsum with or without fiber, but not exceeding 15 per cent of fiber by weight, and surfaced with paper or other fibrous material firmly bonded to the core.

Flexural Strength

3. When tested, either face up or face down, in accordance with the Standard Methods of Testing Gypsum and Gypsum Products (A.S.T.M. Designation: C 26) of the American Society for Testing Materials,[3] specimens taken from the gypsum lath shall carry not less than the following loads without showing a breaking of the bond between the surfacing and the core:

Thickness, in.	Load, lb. Bearing Edges Across Fiber of Surfacing	Load, lb. Bearing Edges Parallel to Fiber of Surfacing
$\frac{1}{4}$	40	16
$\frac{5}{16}$	52	22
$\frac{3}{8}$	60	27
$\frac{1}{2}$	100	40

Sampling

4. At least 1 per cent of the number of gypsum lath in a shipment, but not less than ten lath, shall be so selected as to be representative of the shipment. Twenty-five per cent of such selected lath but not less than ten individual lath, shall constitute a sample for purpose of tests. When a shipment consists of more than one car or carrier load, a sample shall be taken from each car or carrier.

Dimensions, Weights, and Permissible Variations

5. (*a*) *Thickness.*—The nominal thickness of gypsum lath shall be $\frac{1}{4}$ in., $\frac{5}{16}$ in., $\frac{3}{8}$ in., or $\frac{1}{2}$ in., with permissible variations

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-11 on Gypsum.

[2] Prior to their present adoption as standard, these specifications were published as tentative from 1921 to 1925, being revised in 1922 and 1925. They were adopted in 1925, revised 1930, but withdrawn and replaced in 1933 by C 37 – 31 T which was published as tentative from 1931 to 1934.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

in the nominal thickness of plus or minus $\frac{1}{32}$ in., and with permissible local variations of plus or minus $\frac{1}{16}$ in. from the nominal thickness.

(*b*) *Width.*—The nominal width of gypsum lath shall be 16 in., 24 in., or 32 in., with permissible variations of $\frac{1}{4}$ in. under and $\frac{1}{8}$ in. over the specified width.

(*c*) *Length.*—The nominal length of gypsum lath shall be 32 in., 36 in., or 48 in., with permissible variations of $\frac{1}{4}$ in. under and $\frac{1}{8}$ in. over the specified length.

(*d*) *Weight.*—The weight of gypsum lath per 1000 sq. ft. shall conform to the following requirements:

Thickness, in.	Weight per 1000 sq. ft., lb. Minimum	Maximum
$\frac{1}{4}$	900	1500
$\frac{5}{16}$	1125	1750
$\frac{3}{8}$	1350	2000
$\frac{1}{2}$	1800	3000

Finish

6. (*a*) The surfaces of gypsum lath shall be such that they will readily receive and retain gypsum plaster. The edges and ends shall be straight and solid. The corners shall be square with a permissible variation of $\frac{1}{4}$ in. in the full width of the lath. Gypsum lath shall be free of cracks and imperfections that will render them unfit for use.

(*b*) Gypsum lath that have their corners burred or broken shall be considered acceptable provided that the broken portion is not more than 1 in. along either dimension.

NOTE.—Gypsum lath is also available with one surface covered with aluminum or other heat-reflecting type of foil.

Packing and Marking

7. (*a*) Gypsum lath shall be shipped so as to be kept dry and free from moisture.

(*b*) When shipped for resale, the name of the manufacturer and the brand shall be legibly marked on each lath or package.

Inspection

8. Inspection may be made either at the point of shipment or at the point of delivery. The inspector representing the purchaser shall have free access to the carriers being loaded for shipment to the purchaser. He shall be afforded all reasonable facilities for inspection and sampling, which shall be so conducted as not to interfere unnecessarily with the loading of the carriers.

Rejection

9. Any rejection shall be based upon failure to conform to the requirements of these specifications and shall be reported to the seller within 10 working days from receipt of the shipment by the purchaser. The notice of rejection shall contain a specific statement of the respects in which the lath have failed to conform to the requirements of these specifications.

Rehearing

10. Claims for rehearing shall be valid only if made within 20 working days from the receipt of notice of specific cause for rejection.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: A68.1-1942

Standard Specifications for

GYPSUM SHEATHING BOARD[1]

A.S.T.M. Designation: C 79 – 42

ADOPTED, 1934; REVISED, 1942.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 79; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover gypsum sheathing board, which is designed to be used as a sheathing in buildings.

Composition

2. Gypsum sheathing board shall consist of an incombustible core of gypsum with or without fiber, but not exceeding 15 per cent of fiber by weight, and surfaced with paper or other fibrous material firmly bonded to the core.

Flexural Strength

3. When tested, either face up or face down, in accordance with the Standard Methods of Testing Gypsum and Gypsum Products (A.S.T.M. Designation: C 26) of the American Society for Testing Materials,[3] specimens taken from the gypsum sheathing boards shall carry not less than the following loads without showing a breaking of the bond between the surfacing and the core:

Thickness, in.	Load, lb. Bearing Edges Across Fiber of Surfacing	Load, lb. Bearing Edges Parallel to Fiber of Surfacing
$\frac{1}{2}$	110	50

Sampling

4. At least 1 per cent of the number of gypsum sheathing boards in a shipment, but not less than ten boards, shall be so selected as to be representative of the shipment. Twenty-five per cent of such selected boards, but not less than ten individual boards, shall constitute a sample for purpose of tests. When a shipment consists of more than one car or carrier load, a sample shall be taken from each car or carrier.

Dimensions, Weights, and Permissible Variations

5. (*a*) *Thickness.*—The nominal thickness of gypsum sheathing board shall be $\frac{1}{2}$ in., with permissible variations in the nominal thickness of plus or minus $\frac{1}{32}$ in., and with permissible local variations of plus or minus $\frac{1}{16}$ in. from the nominal thickness.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-11 on Gypsum.

[2] Prior to adoption as standard, these specifications were published as tentative from 1930 to 1934, being revised in 1931.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(*b*) *Width.*—The nominal width of gypsum sheathing board shall be 24 in., with a permissible variation of plus or minus $\frac{1}{8}$ in. from the specified width.

(*c*) *Length.*—The nominal length of gypsum sheathing board shall be either 6 ft. 8 in. or 8 ft., with permissible variations of plus or minus $\frac{1}{4}$ in. from the specified length.

(*d*) *Weight.*—The weight of gypsum sheathing board per 1000 sq. ft. shall conform to the following requirements:

Thickness, in.	Weight per 1000 sq. ft., lb. Minimum	Maximum
$\frac{1}{2}$	1800	3000

Finish

6. The edges and ends of gypsum sheathing board shall be straight and solid. The corners shall be square with a permissible variation of $\frac{1}{8}$ in. in the full width of the board. Gypsum sheathing board shall be free of cracks and imperfections that will render them unfit for use.

NOTE.—Gypsum sheathing board is available either with square edges or V-tongue and groove edges.

Packing and Marking

7. (*a*) Gypsum sheathing board shall be shipped so as to be kept dry and free from moisture.

(*b*) When shipped for resale, the name of the manufacturer and the brand shall be legibly marked on each board or package.

Inspection

8. Inspection may be made either at the point of shipment or at the point of delivery. The inspector representing the purchaser shall have free access to the carriers being loaded for shipment to the purchaser. He shall be afforded all reasonable facilities for inspection and sampling, which shall be so conducted as not to interfere unnecessarily with the loading of the carriers.

Rejection

9. Any rejection shall be based upon failure to conform to the requirements of these specifications and shall be reported to the seller within 10 working days from the receipt of the shipment by the purchaser. The notice of rejection shall contain a specific statement of the respects in which the boards have failed to conform to the requirements of these specifications.

Rehearing

10. Claims for rehearing shall be valid only if made within 20 working days from the receipt of notice of specific cause for rejection.

Standard Specifications for

GYPSUM PARTITION TILE OR BLOCK[1]

A.S.T.M. Designation: C 52 – 41

ADOPTED, 1925; REVISED, 1927, 1933, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 52; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover gypsum partition tile or block, a gypsum building unit in form of tile or block for use in non-load-bearing construction in the interior of buildings and for the protection of columns, elevator shafts, etc., against fire.

NOTE 1.—The words "tile" and "block" are synonymous and are used so as to be in accord with certain freight association nomenclature.

NOTE 2.—Gypsum cored tile, 3 or 4 in. in thickness, may be split and used for furring purposes.

Composition

2. Gypsum tile shall consist of set gypsum with or without aggregates. The weight of combustible materials shall not exceed 15 per cent of the weight of the dry tile.

Compressive Strength

3. (*a*) When tested in accordance with the Standard Methods of Testing Gypsum and Gypsum Products (A.S.T.M. Designation: C 26) of the American Society for Testing Materials,[2] the compressive strength of gypsum tile shall be not less than 75 psi., based on gross area.

(*b*) When completely saturated, the compressive strength shall be not less than one third of the strength required for such tile when dried to constant weight.

Sampling

4. At least 1 per cent of the number of gypsum tile in a shipment, but in no case less than ten tile, shall be so selected as to be representative of the shipment. Twenty-five per cent of such selected tile, but in no case less than ten tile, shall constitute a sample for purpose of tests. In case a shipment consists of more than one car or carrier load, a sample shall be selected from each car or carrier. Samples for test shall be selected at the place of manufacture or at the point of delivery, as specified in the order.

Form

5. Gypsum tile may be solid or cored and shall be rectangular in shape with

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-11 on Gypsum.

[2] Prior to adoption as standard, these specifications were published as tentative from 1923 to 1925, being revised in 1924 and 1925.

[2] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

straight and square edges and true surfaces. They may also be of special shape, provided the requirements as otherwise prescribed in these specifications are met. They shall be free from cracks and other imperfections that would render them unfit for use and shall be dried before shipment.

Core Spaces

6. (*a*) The core spaces shall be symmetrically spaced.

(*b*) *Shell Thickness.*—Shell thickness shall be not less than the following:

Thickness of Tile, in.	Side and Edge Shell Thicknesses, in. Circular Cores	Elliptical or Rectangular Cores
3	$\frac{1}{2}$	$\frac{11}{16}$
4	$\frac{1}{2}$	$\frac{13}{16}$
5	$\frac{7}{16}$	1
6	$\frac{1}{2}$	$1\frac{1}{16}$

The sum of the thicknesses of the two side (face) shells plus the thickness of the center vertical web of 6-in. tile shall be not less than 2 in. for tile having circular cores and not less than $2\frac{3}{4}$ in. for tile having elliptical or rectangular cores.

Dimensions

7. (*a*) Gypsum tile may be of any convenient length and height, but in general they shall not exceed 30 in. in length and 12 in. in height. The thickness shall be uniform throughout and within the limits specified in Paragraph (*b*).

(*b*) *Thickness.*—Gypsum tile shall conform to the specified thicknesses within the following permissible variations:

Nominal Size of Tile or Block, in.	Minimum Thickness, in.	Maximum Thickness, in.
$1\frac{1}{2}$ (Furring)	$1\frac{3}{8}$	$1\frac{5}{8}$
2 (Furring)	$1\frac{3}{8}$	$2\frac{1}{8}$
2	2	$2\frac{1}{8}$
3	$2\frac{7}{8}$	$3\frac{1}{8}$
4	$3\frac{7}{8}$	$4\frac{1}{8}$
5	$4\frac{7}{8}$	$5\frac{1}{8}$
6	$5\frac{7}{8}$	$6\frac{1}{8}$

(*c*) *Measurement of Dimensions.*—Over-all dimensions shall be measured between opposite faces of the tile, the greatest measurement observed being taken as the dimension.

Sectional dimensions of cored tile shall be taken 1 in. in from the ends of the tile, the least measurement observed being taken as the dimension.

Shell dimensions shall be measured from the surface of the core space to the normal surface of the tile inside the scoring.

(*d*) *Permissible Variations in Dimensions.*—A variation of $\frac{1}{4}$ in. in the nominal height and $\frac{3}{8}$ in. in the nominal length of the tile is permissible.

Scoring

8. When the surfaces of the tile are scored, the scoring shall not reduce materially the thickness of the shell. Surfaces of the tile shall be such that they afford a suitable bond with plaster.

Marking

9. When gypsum tile are shipped for resale, the manufacturer's "Brand" name shall be shown upon each tile.

Inspection

10. Inspection may be made either at the place of manufacture or at the point of delivery. The inspector representing the purchaser shall have free access to the carriers being loaded for shipment to the purchaser. He shall be afforded all reasonable facilities for inspection and sampling, which shall be so conducted as not to interfere unnecessarily with the loading of the carriers.

Rejection

11. Any rejection shall be based upon

failure to conform to the requirements of these specifications, and shall be reported within 10 working days from the receipt of the shipment by the purchaser. The notice of rejection shall contain a specific statement of the respects in which the tile have failed to meet the requirements of these specifications.

Rehearing

12. Claims for rehearing shall be valid only if made within 20 working days from receipt of notice of specific cause for rejection.

APPROVED AS
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ASA NO.: A70.1-1942

Standard Methods of

TESTING GYPSUM AND GYPSUM PRODUCTS[1]

A.S.T.M. Designation: C 26 - 42

ADOPTED, 1923; REVISED, 1927, 1930, 1933, 1939, 1940, 1942.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 26; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods cover procedures for the chemical analysis and physical testing of gypsum and gypsum products.

Free Water

2. (*a*) Weigh a sample of not less than 1 lb. of the material as received and spread it out in a thin layer in a suitable vessel. Place in an oven and dry at 113 F. (45 C.) for 2 hr., then cool in an atmosphere free from moisture, and weigh again. The loss of weight corresponds to the free water, and shall be calculated as a percentage of the sample as received.

(*b*) Retain the dried sample in an air-tight container until used for the fineness test (Section 3).

Fineness

3. Determine fineness by sieving a known weight of the dried sample through sieves of the specified sizes (Note). The size of the sample to be used in determining fineness depends upon the particle size of the material. If the material will pass a $\frac{1}{4}$-in. sieve,[3] a 100-g. sample will be sufficient; if the largest particles are more than 1 in. in diameter, use at least a 1000-g. sample. With these limitations the size of sample to be used is left to the discretion of the operator. Shake the sample through each sieve with as little abrasion as possible. Weigh the amount of material retained on each sieve and calculate the fineness expressed as a percentage of the weight of the original sample.

NOTE.—The sizes of the sieves to be used are given in the specifications of the American Society for Testing Materials covering the particular product in question.

CHEMICAL ANALYSIS

Preparation of Sample

4. Weigh a sample of not less than 1 lb. of the material as received and spread it out in a thin layer in a suitable vessel. Place in an oven and dry at 113 F. (45 C.) for 2 hr., then cool in an

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee C-11 on Gypsum.

[2] Prior to adoption as standard, these methods were published as tentative from 1919 to 1923, being revised in 1920, 1921, and 1923.

[3] Detailed requirements for this sieve are given in the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

A "tentative revision" of this standard appears in the Tentative Revisions of Standards section at the back of this book.

atmosphere free from moisture. Reduce the sample to pass a No. 60 (250-micron) sieve,[3] using extreme care not to expose unduly the material to moisture or to overheating. Thoroughly remix the ground sample, and store it in an air-tight container until used.

COMPLETE PROCEDURE

Combined Water

5. Place 1 g. of the sample in a covered crucible and dry to constant weight at 420 to 445 F. (215 to 230 C.). Calculate the loss of weight to percentage of sample as received, and report as combined water.

Carbon Dioxide

6. Place the residue, obtained after drying as described in Section 5, in a suitable flask and dissolve it in diluted HCl (not stronger than 1:4) in such a way that the gas evolved, after being freed from water vapor by calcium chloride or sulfuric acid, can be collected in either soda-lime or caustic potash and weighed. Boil the solution for 1 min., and pass a current of CO_2-free air through the apparatus for 30 min. The increase of weight of the soda-lime or caustic potash corresponds to the weight of carbon dioxide. Calculate the CO_2 to percentage of sample as received.

Silicon Dioxide and Insoluble Matter

7. Place 0.5 g. of the sample in a porcelain casserole. Add about 25 ml. of HCl (1:5), and evaporate to apparent dryness on a hot plate. Cool, and add enough HCl (sp. gr. 1.19) to wet thoroughly. Add about 10 ml. of water, boil, filter, and wash. Put the filtrate back in the same casserole. Evaporate it to dryness and heat to about 250 F. (120 C.) for 1 hr. and then cool. Add enough HCl (sp. gr. 1.19) to wet thoroughly. Add about 25 ml. of water, boil, filter, and wash. Transfer the two papers containing the two precipitates to the same crucible, ignite, and weigh. Calculate the SiO_2 and insoluble matter to percentage of sample as received.

Iron and Aluminum Oxides

8. To the filtrate obtained as described in Section 7, add a few drops of HNO_3, and boil to insure oxidation of the iron. Add 2 g. of NH_4Cl previously dissolved in water. Make alkaline with NH_4OH. Digest hot for a few minutes until the precipitate coagulates. Filter, wash, ignite the precipitate, and weigh as $Fe_2O_3 + Al_2O_3$. Calculate the iron and aluminum oxides to percentage of sample as received. This precipitate may be further treated to separate the two oxides, but this is generally unnecessary.

Lime

9. (*a*) To the filtrate obtained as described in Section 8, add 5 g. of $(NH_4)_2C_2O_4$ dissolved in water. Digest hot for 30 min., making sure that the solution is always alkaline with NH_4OH. Filter, wash, and ignite to constant weight in a platinum crucible over a strong blast. Calculate the CaO to percentage of sample as received.

(*b*) *Alternative Method.*—To the filtrate obtained as described in Section 8, add 5 g. of $(NH_4)_2C_2O_4$ dissolved in water. Digest hot for 30 min., making sure that the solution is always alkaline with NH_4OH. Filter and wash. Transfer the precipitate to a beaker, and wash the filter paper with hot diluted H_2SO_4, catching the washings in the same beaker. Heat gently to complete solution, adding more H_2SO_4 if necessary. While still warm, titrate with a solution of $KMnO_4$ containing 5.6339 g. per liter, until the pink color is permanent. The number of milliliters of $KMnO_4$ used gives directly the percentage of lime in the dried sample.

Recalculate the CaO to percentage of sample as received.

Magnesium Oxide

10. To the filtrate obtained as described in Section 9 (*a*) or (*b*), add enough water to give a total volume of about 600 ml. Cool, and add 10 ml. of NH_4OH and 5 g. $NaNH_4HPO_4$ dissolved in water. Stir until precipitate begins to form. Let stand over night. Filter, wash with NH_4NO_3 (2.5 per cent). Ignite and weigh. Multiply this weight by 0.36207 to find the weight of MgO. Calculate the MgO to percentage of sample as received.

Sulfur Trioxide

11. Dissolve 0.5 g. of the sample in 50 ml. of HCl (1:5). Boil. Add 100 ml. of boiling water, and continue boiling for 5 min. Filter immediately and wash thoroughly with hot water. Boil, and while boiling, add slowly 20 ml. of a boiling solution of $BaCl_2$ (10 per cent). Digest hot for 1 hr., or until the precipitate settles. Filter and wash. Dry carefully. Ignite over a bunsen burner at the lowest heat possible until the filter paper is burned off. Ignite at bright red heat for 15 min., and weigh. Multiply this weight by 0.34297 to determine the weight of SO_3. Calculate the SO_3 to percentage of sample as received.

Sodium Chloride

12. Dissolve 1 g. of the sample, in boiling water, filter, and wash with 250 ml. of boiling water. Add two or three drops of K_2CrO_4 to the filtrate and titrate with $AgNO_3$ (0.05 *N*). One milliliter of $AgNO_3$ is equivalent to 0.002923 g. of sodium chloride. Calculate the NaCl to percentage of sample as received.

Report

13. Report the results obtained in the analysis as follows:

	Per cent
Free water	.....
Combined water	.....
Carbon dioxide, CO_2	.....
Silicon dioxide (SiO_2) and insoluble matter	.....
Iron and aluminum oxides, $Fe_2O_3 + Al_2O_3$	.....
Lime, CaO	.....
Magnesium oxide, MgO	.....
Sulfur trioxide, SO_3	.....
Sodium chloride, NaCl	.....
Total	100.00±

NOTE 1.—Since it is frequently advisable to recalculate the results obtained in the chemical analysis in order that they may be more enlightening, the following is submitted for consideration:

(*a*) Multiply percentage of MgO by 2.0912 to find percentage of $MgCO_3$.

(*b*) Multiply the percentage of MgO by 1.0914 to find the percentage of CO_2 as $MgCO_3$.

(*c*) Deduct CO_2 as $MgCO_3$ from the CO_2 determined.

(*d*) Multiply the CO_2 remaining by 2.2742 to find percentage of $CaCO_3$.

(*e*) Add together the percentage of SiO_2, $Fe_2O_3+Al_2O_3$, $MgCO_3$, and $CaCO_3$, and report in the aggregate.

(*f*) Multiply the percentage of $CaCO_3$ by 0.56031 to find the percentage of CaO as $CaCO_3$.

(*g*) From the total percentage of CaO, deduct the percentage of CaO as $CaCO_3$. The remainder may be called "available CaO."

(*h*) The "available CaO" should bear to the SO_3 a ratio of 0.6991 to 1. Determine which (if either) is in excess.

(*i*) If the CaO is in excess, multiply the SO_3 by 0.6991, and subtract the result from the "available CaO." The remainder is reported as "excess CaO."

(*j*) If the SO_3 is in excess, multiply the "available CaO" by 1.4304 and subtract the result from the SO_3. The remainder is reported as "excess SO_3."

(*k*) Add together the "available CaO," and the SO_3, and subtract the "excess CaO" or "excess SO_3." The remainder is $CaSO_4$.

(*l*) If the $CaSO_4$ is present as $CaSO_4 \cdot \frac{1}{2}H_2O$, the percentage of $CaSO_4$ should bear to the percentage of combined water a ratio of 15.12 to 1. Determine which (if either) is in excess.

(*m*) If the $CaSO_4$ is in excess, some of it is present in the anhydrous form. Multiply the

percentage of combined water by 15.12 to find the percentage of $CaSO_4$ as $CaSO_4 \cdot \frac{1}{2}H_2O$. The difference between the total $CaSO_4$ and the percentage of $CaSO_4$ as $CaSO_4 \cdot \frac{1}{2}H_2O$, is the $CaSO_4$ in the anhydrous form.

(*n*) If the water is in excess, some of the $CaSO_4$ is present as gypsum. Let x = percentage of $CaSO_4 \cdot \frac{1}{2}H_2O$, and y = percentage of $CaSO_4 \cdot 2H_2O$.

Then:

$x + y$ = percentage of $CaSO_4$ (as found in *k*) + percentage of water.

$0.06206x + 0.2093y$ = percentage of combined water.

Solve these equations for x and y. Report x as percentage of "calcined gypsum," $CaSO_4 \cdot \frac{1}{2}H_2O$. Report y as percentage of gypsum, $CaSO_4 \cdot 2H_2O$.

NOTE 2.—Having made the calculations listed in Note 1, the results may be reported as follows:

	Per cent
Gypsum, $CaSO_4 \cdot 2H_2O$	.....
Calcined gypsum, $CaSO_4 \cdot \frac{1}{2}H_2O$	.....
Anhydrite $CaSO_4$	.....
Excess CaO or Excess SO_3	.....
Sodium chloride, NaCl	.....
Other ingredients	.....
Total	100.00±

NOTE 3.—The presence of the different forms of $CaSO_4$ may be corroborated by a microscopic examination.

ALTERNATIVE PROCEDURE FOR CALCIUM SULFATE BY AMMONIUM ACETATE METHOD[4]

Nature of Test

14. This procedure is intended for determining calcium sulfate in gypsum and gypsum products by extraction with ammonium acetate solution, and may be used as an alternative method to the complete procedure described in Sections 2 to 13.

Reagents

15. (*a*) *Ammonium Acetate Solution.*—Dissolve 454 g. of c.p. ammonium acetate in 2 liters of water. Add sufficient NH_4OH to make the solution distinctly ammoniacal, using phenolphthalein as the indicator.

(*b*) *Ammonium Hydroxide Wash Solution.*—Dilute 100 ml. of NH_4OH (sp. gr. 0.90) to 1 liter.

(*c*) *Filter Aid.*—Analytical grade diatomaceous silica.

Procedure Using Gooch Crucible

16. (*a*) Weigh rapidly approximately 4 g. (Note 1) of the well-mixed sample and transfer to a 600-ml. beaker.

NOTE 1.—All weighings shall be made to 0.001 g., except that weights of crucibles and of their contents shall be determined to 0.0001 g.

(*b*) Without delay, weigh 1 g. of the well-mixed sample in a tared weighing bottle having a ground-glass stopper. Dry the sample and weighing bottle to constant weight at 45 C. (Note 2), and calculate the percentage loss in weight at 45 C. (largely uncombined water) as follows:

$$\text{Loss in weight at 45 C., per cent} = \frac{A - B}{C} \times 100$$

where:

A = original weight of sample and weighing bottle,

B = weight of sample and weighing bottle dried to constant weight at 45 C., and

C = original weight of sample.

Calculate the weight of the 4-g. sample (Paragraph (*a*)) corrected for loss on heating to constant weight at 45 C.

If the percentage by weight of combined water held by the calcium sulfate is desired, heat the sample and weighing bottle to constant weight at 220 C. (Note 2), and calculate the percentage of combined water as follows:

$$\text{Combined water, per cent} = \frac{B - D}{B - E} \times 100$$

[4] This procedure was prepared by L. S. Wells and W. F. Clarke, National Bureau of Standards, and modified by B. E. Kester, United States Gypsum Co.

where:

B = weight of sample and weighing bottle dried to constant weight at 45 C.,
D = weight of sample and weighing bottle dried to constant weight at 220 C., and
E = weight of weighing bottle.

NOTE 2.—Stopper weighing bottles immediately upon removal from the oven in order to prevent absorption of moisture from the air upon cooling.

(*c*) To the contents of the 600-ml. beaker (Paragraph (*a*)), add 350 ml. of the ammonium acetate solution, and stir the mixture thoroughly so as to loosen all of the solid matter from the bottom of the beaker. Add 0.2000 g. of redried diatomaceous silica to the mixture, heat the beaker and contents to 70 C. on a steam bath, and maintain at that temperature for 30 min., while stirring frequently. During the heating, keep the solvent ammoniacal by additions of NH_4OH and phenolphthalein, if indicated. (Meanwhile, heat a supply of the ammonium acetate solution to 70 C., keeping it also distinctly ammoniacal.) Filter the mixture, with suction, through a tared Gooch crucible, stirring frequently during filtration to keep the diatomaceous earth suspended in the liquid. Wash the Gooch crucible containing the residue with five 10-ml. portions of the warm acetate solution, draining thoroughly after each washing. Wash in the same manner with eight 10-ml. portions of the NH_4OH wash solution. Care shall be exercised to wash the upper walls of the Gooch crucible. Drain the crucible dry with suction, place in an oven at 70 C., and dry to constant weight (Note 3). Allow the crucible to cool in a desiccator before weighing.

NOTE 3.—Avoid overheating in all oven drying of ammonium acetate residues; that is, place crucibles well away from the heating elements. This is of particular importance for samples high in impurities, as these impurities often have water of hydration that is lost on local overheating.

(*d*) Calculate the percentage of $CaSO_4 \cdot XH_2O$ on the basis of the sample dried to constant weight at 45 C., as follows:

$$CaSO_4 \cdot XH_2O, \text{ per cent} = \frac{F - (G - H)}{F} \times 100$$

where:

F = weight of sample, corrected for loss on heating to constant weight at 45 C.,
G = weight of dried crucible and contents, and
H = weight of crucible plus diatomaceous silica used as filter aid.

Procedure Using Tared Filter Papers[5]

17. (*a*) Dry a quarter-folded, 11-cm., quantitative filter paper overnight at 70 C. in a wide-form, glass-stoppered, 30 by 60-mm. weighing bottle. After drying, cool the weighing bottle and paper in a desiccator, and weigh.

(*b*) Treat the sample exactly as described in Section 16 prior to the filtration. Filter the mixture by gravity through an ordinary 7-cm. glass funnel, stirring frequently during filtration to keep the diatomaceous silica suspended in the liquid. Wash the filter paper and residue with five 10-ml. portions of warm acetate solution, draining thoroughly after each washing. Wash in the same manner with eight 10-ml. portions of the NH_4OH wash solution. After final draining replace the paper and residue in the weighing bottle, and dry at 70 C. to constant weight (see Note

[5] This procedure is suggested where several samples are to be analyzed at once. It has been found that gravity filtration on six samples will proceed as rapidly as it is possible to handle them.

3 under Section 16 (*c*)). Cool the weighing bottle, paper, and residue in a freshly prepared desiccator before weighing; this is essential, due to the hygroscopic character of paper.

(*c*) Calculate the percentage of $CaSO_4 \cdot XH_2O$ as described in Section 16 (*d*), substituting the weight of the weighing bottle and filter paper for the weight of the crucible.

PHYSICAL TESTS

Precautions for Physical Tests

18. Gypsum products are peculiar in that their properties are very greatly affected by the small amounts of impurities which may be introduced by careless laboratory manipulation. In order to obtain concordant results, it is therefore absolutely essential to observe the following precautions:

(*a*) All apparatus shall be kept thoroughly clean. Especially shall all traces of set plaster be removed.

(*b*) Distilled water, free from chlorides and sulfates, shall be used exclusively.

(*c*) Standard sand shall be used exclusively consisting of a natural silica sand from Ottawa, Ill. It shall be prepared for use by washing once with HCl (1:4) and four times with distilled water, and then drying.

NORMAL CONSISTENCY OF GYPSUM PLASTER

NOTE.—An accurate method for determining normal consistency is the most important step in the standardizing of physical methods for testing cementitious materials.

Apparatus

19. (*a*) *Modified Vicat Apparatus.*—The modified Vicat apparatus (Fig. 1) shall consist of a bracket, *A*, bearing a movable brass rod, *B*, 6.3 mm. in diameter and of suitable length to fit the Vicat bracket. On the lower end of the rod shall be attached a plunger, *C*, 19.0 mm. in diameter and 44.4 mm. in length, made of aluminum tubing. The total weight of the rod with plunger shall be 50 g. This total weight may

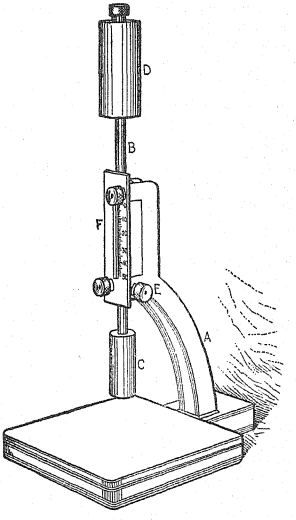


FIG. 1.—Modified Vicat Apparatus.

be increased by means of a weight, *D*, screwed into the rod. The rod can be held in any desired position by a screw *E*. The rod shall have a mark midway between the ends which moves under a scale, *F*, graduated in millimeters, attached to the bracket, *A*.

(*b*) *Mold.*—The conical ring mold shall be made of a noncorroding, nonabsorbent material, and shall have an inside diameter of 7 cm. at the base and 6 cm. at the top and a height of 4 cm.

(*c*) *Base Plate.*—The base plate for supporting the ring mold shall be of plate glass and about 10 cm. square.

Procedure

20. (*a*) The plunger, mold, and base plate of the modified Vicat apparatus shall be cleaned. A thin coat of vaseline or other suitable lubricant shall be applied to the upper surface of the base plate in order to prevent leaks during the test.

(*b*) A 200-g. sample shall be sifted into a known volume of water to which has been previously added 0.2 g. of commercial retarder. After allowing the sample to soak for 2 min. the mixture shall be stirred for 1 min. to an even fluidity. This sample shall then be poured into the ring mold, worked slightly to remove air bubbles and then struck off flush with the top of the mold. The plunger of the modified Vicat apparatus shall then be wet and lowered to the surface of the sample at approximately the center of the mold. The scale shall be read and the plunger released immediately. After the rod has settled, the scale shall be read again. Readings are reproducible on a retarded mix, and therefore, in order to eliminate error, two or three determinations should be made on each mix, care being taken to have the mold completely filled and the plunger clean and wet.

Normal Consistency

21. (*a*) *Normal Consistency, Calcined Gypsum.*—Calcined gypsum shall be considered of normal consistency when a penetration of 30 ± 2 mm. is obtained when tested in accordance with Section 20, weight of rod and plunger for this determination to be 50 g. Normal consistency shall be expressed as the number of milliliters of water required to be added to 100 g. of the gypsum.

(*b*) *Normal Consistency, Gypsum Mixtures.*—Sanded gypsum mixtures and gypsum wood-fiber plaster shall be considered of normal consistency when a penetration of 20 ± 3 mm. is obtained when tested in accordance with Section 20, weight of rod and plunger for these determinations to be 150 g. Normal consistency shall be expressed as the number of milliliters of water required to be added to 100 g. of the mixture.

WATER-CARRYING CAPACITY

Water-Carrying Capacity

22. The water-carrying capacity is the amount of water, calculated as percentage by weight of the dry material, that is necessary to bring the mixture to normal consistency.

DRY BULK

Procedure

23. Dry bulk is the volume in cubic centimeters occupied by 100 g. of dry material.[6] A No. 10 (2000-micron) sieve[3] shall be placed above a No. 20 (840-micron) sieve[3] and the two sieves held at a height of 12 in. (30 cm.) above a perpendicular-sided receptacle of known volume (about 400 cu. cm.). The material under test shall be placed on top of the No. 10 sieve and passed through the No. 10 and No. 20 sieves, filling the receptacle. The material shall be struck off flush with the top of the receptacle and the net weight obtained of the contents in grams. The average result of three tests shall be obtained according to this method and reported in cubic centimeters per 100 g.

WET BULK

Wet Bulk

24. Wet bulk is the volume in cubic centimeters occupied by a mixture of

[6] Dry bulk is expressed as the volume in cubic centimeters occupied by 100 g. of dry material instead of "grams per 100 cu. cm.," since bulk is understood to mean volume per unit weight rather than weight per unit volume.

100 g. of dry material mixed with water to normal consistency.

TIME OF SETTING

Apparatus

25. (*a*) *Vicat Apparatus.*—The Vicat apparatus (Fig. 2) shall consist of a frame *A*, bearing a movable rod *B*, weighing 300 g., one end *C*, the plunger end, being 1 cm. in diameter for a distance of at least 5 cm., the other end having a removable needle *D*, 1 mm. in diameter and 5 cm. in length. The rod *B* shall be reversible, and can be held in any desired position by a screw *E*, and shall have an adjustable indicator *F* which moves over a scale (graduated in millimeters) attached to the frame *A*. The paste is held in a rigid conical ring *G*, resting on a glass plate *H* about 10 cm. square. The ring shall be made of a noncorroding, nonabsorbent material and shall have an inside diameter of 7 cm. at the base, 6 cm. at the top, and a height of 4 cm.

(*b*) In addition to the above, the Vicat apparatus shall conform to the following requirements:

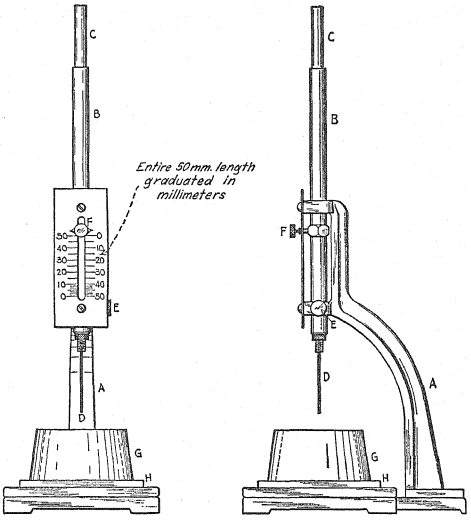


FIG. 2.—Vicat Apparatus.

Weight of plunger	300 ± 0.5 g. (0.661 lb. ± 3 grains)
Diameter of larger end of plunger.	1 ± 0.005 cm. (0.394 ± 0.002 in.)
Diameter of needle	1 ± 0.05 mm. (0.039 ± 0.002 in.)
Inside diameter of ring at bottom	7 ± 0.3 cm. (2.75 ± 0.12 in.)
Inside diameter of ring at top	6 ± 0.3 cm. (2.36 ± 0.12 in.)
Height of ring.	4 ± 0.1 cm. (1.57 ± 0.04 in.)
Graduated scale	The graduated scale, when compared with a standard scale accurate to within 0.1 mm. at all points, shall not show a deviation at any point greater than 0.25 mm.

Procedure

26. (*a*) *All Calcined Gypsum Products, Except Gypsum Neat Plaster.*—A 200-g. sample (Note) shall be mixed with enough water to make a paste of normal consistency as described in Sections 19 to 21. The paste shall be placed in the conical ring and, after completely filling the ring, the paste shall be tested for final set with a Vicat needle. When conducting the test, the needle shall be allowed to sink into the paste at frequent intervals. After each penetration, the needle shall be wiped clean, and the paste moved slightly so that the needle will not strike the same place twice. The frequency of the penetration will depend upon the character of the material. If the sample is not retarded, it shall be tested every 2 min. until nearly set, and then every 1 min. If the sample is retarded, it shall be tested at such intervals as are necessary to determine whether it complies with the requirements for time of setting for the product tested. Set shall be considered complete when the needle no longer penetrates to the bottom of the paste. The elapsed time in minutes from the time when the sample was first added to the water to the time when set is complete shall be recorded as the time of setting of the sample.

NOTE.—When the material to be tested is gypsum ready-mixed sanded plaster, a 300-g. sample shall be mixed to normal consistency for sanded gypsum mixture.

(*b*) *Gypsum Neat Plaster.*—Gypsum neat plaster shall be tested for time of setting as mixed with three parts by weight of standard sand that has been washed and dried in accordance with the procedure described in Section 18 (*c*). A 100-g. sample of the gypsum neat plaster and 300 g. of the standard sand shall be mixed dry, and then sufficient water added to produce a mixture of normal consistency for sanded gypsum mixtures as described in Section 21 (*b*). The mortar shall be placed in the conical rings and tested for time of setting as described in Paragraph (*a*). Until set, the test specimens shall be stored in a cabinet at a temperature of not less than 68 F. (20 C.) nor more than 72 F. (22.2 C.) in an atmosphere having a relative humidity of not less than 85 nor more than 100 per cent. The specimens shall not be tested with the Vicat needle until they show evidence of stiffening or setting. Excessive penetration of the mix with the needle tends to accelerate the setting time. For plaster setting in less than 5 hr. the setting time shall be determined to the nearest 15-min. interval, and for plaster setting in more than 5 hr., to the nearest 30-min. interval.

TENSILE STRENGTH

Apparatus

27. (*a*) *Molds.*—The molds for making test specimens shall be made of noncorroding material and shall have sufficient material in the sides to prevent spreading during molding. The dimensions of the briquet molds shall conform to the following requirements: width of mold, between inside faces, at waist line of briquet, 1 in. with permissible variations of plus or minus 0.01 in. for old molds and plus or minus 0.005 in. for new molds; thickness of new mold, measured at point of greatest thickness on either side of mold at waist line, 1 in. with permissible variations of plus 0.004 in. and minus 0.002 in.

(*b*) *Testing Machine.*—Any form of universal testing machine suitable for making tension tests may be used.

Test Specimen

28. (*a*) *All Calcined Gypsum Products Except Gypsum Neat Plaster.*—A

500-g. sample (Note) shall be mixed to normal consistency in accordance with Sections 19 to 21 and cast in a briquet mold of the shape and size shown in Fig. 3. Before being filled, the molds shall be oiled with a thin film of mineral oil. The briquet specimens shall not be cast successively, but instead, the containing vessel shall be moved back and forth over the molds while pouring continuously. The briquets shall be worked slightly with the point of the

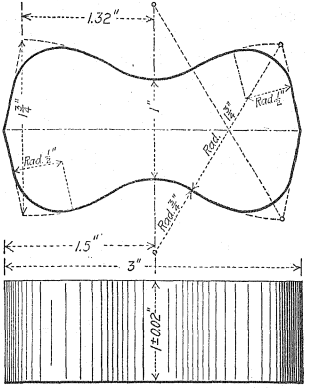


FIG. 3.—Briquet Specimen for Tensile Strength Test.

trowel to remove air bubbles and leveled off flush with the top of the mold. The briquet shall be retained not less than 24 hr. in the mold in moist air until thoroughly hardened. The specimens shall then be removed from the mold and dried at not less than 70 F. (21.1 C.) nor more than 100 F. (37.8 C.) in an atmosphere having a relative humidity of not more than 50 per cent. The specimen shall be weighed at 1-day intervals until the weight has become constant to within 0.1 per cent.

NOTE.—When the material to be tested is gypsum ready-mixed sanded plaster, a sample of not less than 750 g. shall be used and mixed to normal consistency for sanded gypsum mixture.

(*b*) *Gypsum Neat Plaster.*—Gypsum neat plaster, both fibered and unfibered, shall be tested for tensile strength as mixed with two parts of standard sand; fibered plaster shall be sieved through a No. 6 (3360-micron) sieve to remove the major portion of the long fibers present. A 250-g. sample of the gypsum neat plaster and 500 g. of standard sand shall be mixed dry, and then sufficient water added to produce a mixture of normal consistency in accordance with Section 21 (*b*). The mortar shall be cast into the molds as described in Paragraph (*a*), except that the molds shall be so filled as to allow a slight excess of the mortar to extend above the top of the molds. After the mortar has set, the excess mortar shall be screeded from the top of the molds with a broad knife or similar implement. The mortar briquet specimens shall be dried as described in Paragraph (*a*).

Procedure

29. As soon as the briquet specimens have been dried (Section 28) their tensile strengths shall be determined.

Report

30. The average tensile strength shall be reported as the tensile strength of the material, except that if the strengths of one or two of the briquets vary more than 15 per cent from the average of the five, they shall be discarded and the tensile strength shall be reported as the average of the remaining specimens. In case the tensile strengths of three or more briquet specimens vary more than 15 per cent from the average, the results shall be discarded and the test repeated.

COMPRESSIVE STRENGTH

Test Specimens

31. A 1500-g. sample shall be mixed to normal consistency as described in Sections 19 to 21, and cast into five 2-in. split-cube molds set plumb upon metal or glass plates. The cubes shall not be cast successively, but instead, the containing vessel shall be moved back and forth over the molds while pouring continuously. The cubes shall be worked slightly with the point of the trowel to remove air bubbles, and leveled off flush with the top of the molds. The cubes shall be retained not less than 24 hr. in the molds in moist air until thoroughly hardened. The specimens shall then be removed from the molds and dried at a temperature of not less than 70 F. (21.1 C.) nor more than 100 F. (37.8 C.) in an atmosphere having a relative humidity of not more than 50 per cent. The specimens shall be weighed at 1-day intervals until the weight has become constant to within 0.1 per cent.

Procedure

32. As soon as the cube specimens have been dried (Section 31) their compressive strengths shall be determined.

Report

33. The average compressive strength shall be reported as the compressive strength of the material, except that if the strengths of one or two of the cubes vary more than 15 per cent from the average of the five, they shall be discarded and the compressive strength shall be reported as the average of the remaining specimens. In case the tensile strengths of three or more cubes vary more than 15 per cent from the average, the results shall be discarded and the test repeated.

DETERMINATION OF SAND IN SET PLASTER

Ammonium Acetate Method

34. Any method for the determination of the sand content of set gypsum plaster requires for accurate results the following determinations:

(*a*) Determination of the percentage of insoluble matter in the sand used with the plaster,

(*b*) Determination of the percentage of insoluble matter in the gypsum neat plaster, and

(*c*) Determination of the percentage of insoluble matter in the sanded calcined plaster.

NOTE.—If samples of the original gypsum neat plaster and the sand are not available, an approximation of the insoluble matter may be obtained by use of this method on plaster and sand from the same sources as those from which the plaster to be analyzed was originally prepared.

Sampling

35. Where plaster to be tested is part of a two-coat or three-coat plastering operation, the sample for analysis shall be taken from that portion of the entire plaster sheet which comprises the single coat being tested. Succeeding coats of plaster shall be separated by use of a stiff putty knife or similar implement. At least 500 g. shall be taken as a sample, the sample preferably being obtained from different sections of the wall or ceiling under examination.

Procedure

36. (*a*) In a clean porcelain mortar, grind the set-plaster sample to the size of the largest sand particles present, or smaller, so that approximately 100 per cent of the sample will pass a No. 8 (2380-micron) sieve. Fine grinding makes solution of the gypsum faster. Place about 200 g. of the ground sample in a porcelain casserole or evaporating dish, and calcine on a sand bath. Stir

the sample continuously with a thermometer during the heating, and adjust the rate of heating so that 20 to 30 min. will be required to raise the temperature of the sample to 320 ± 9 F. (160 ± 5 C.). Cool the sample to room temperature.

(*b*) After cooling, weigh accurately 20 ± 0.05 g. of the calcined sample into a 600-ml. beaker. Add 300 to 350 ml. of ammonium acetate (25 per cent), which should be slightly alkaline to litmus paper. If acidic, add a few milliliters of diluted NH_4OH (1 per cent) to the stock solution of ammonium acetate to render it slightly alkaline prior to the addition to the test sample.

(*c*) Warm the suspension to a temperature of 158 ± 9 F. (70 ± 5 C.) and stir continuously for 20 to 30 min. Filter the warm suspension with the aid of suction through a small Büchner funnel or Gooch crucible in which an asbestos mat or filter paper has previously been formed, the funnel and mat having been dried at 230 F. (110 C.) to constant weight within 0.01 g. Refilter the first 100 ml. of the filtrate. Wash the sand remaining in the beaker onto the filter with an additional 100 ml. of warm ammonium acetate solution. Wash the beaker and residue with 200 to 300 ml. of water, dry the funnel and sand at 212 F. (100 C.) to constant weight. Subtract the weight of the residue from the original weight of the sample to obtain the weight of the insoluble matter.

(*d*) *Percentage of Insoluble Matter in Plaster.*—Multiply the weight of the insoluble matter obtained in Paragraph (*c*) by 5 to obtain the percentage of insoluble matter in the sanded plaster.

(*e*) *Percentage of Insoluble Matter in Sand.*—Determine the weight of insoluble matter in the sand as described in Paragraphs (*a*) to (*c*), except that no grinding of the sample is necessary. Multiply the weight of the insoluble matter obtained by 5 to obtain the percentage of insoluble matter in the sand.

(*f*) *Percentage of Insoluble Matter in Gypsum Neat Plaster.*—Determine the weight of insoluble matter in the gypsum neat plaster as described in Paragraphs (*a*) to (*c*), except that only a 5-g. sample is required and no grinding of the sample is necessary. Multiply the weight of the insoluble matter obtained by 20 to obtain the percentage of insoluble matter in the gypsum neat plaster.

Calculation

37. (*a*) The percentage of sand in the sanded plaster shall be calculated as follows:

$$X = \frac{(C - B)100}{A - B}$$

where:

X = percentage of sand in sanded plaster,
A = percentage of insoluble matter in the sand,
B = percentage of insoluble matter in the gypsum neat plaster, and
C = percentage of insoluble matter in the sanded plaster.

(*b*) To express the results as a ratio of the parts of sand per part of plaster by weight, the following formula may be used:

$$\text{Ratio of sand to plaster} = \frac{X}{100 - X}$$

NOTE.—The results obtained by the above procedure indicate the amount of sand originally mixed with the gypsum neat plaster before it had been gaged with water or set.

WOOD-FIBER CONTENT IN WOOD-FIBER GYPSUM PLASTER

Procedure

38. (*a*) A 100-g. sample of wood-fiber plaster, prepared as described in Section 4

shall be placed on a No. 30 (590-micron) sieve nested over a No. 100 (149-micron) sieve. The plaster on the No. 30 sieve shall then be washed with a stream of cold water, removing the No. 30 sieve when the fiber on it is practically or entirely free of plaster. The material on the No. 100 sieve shall next be washed until the bulk of the plaster has been washed through the sieve and the residue is mainly fiber. The material retained on the No. 100 sieve shall then be transferred to a 3-qt. vitreous enamel lipped pan, adding the charge on the No. 30 sieve if the fiber contains any adhering particles of plaster. The material in the pan shall then be elutriated (purified by washing and straining, effecting as clean a separation of fiber from plaster as is feasible) catching the elutriated fibers on a No. 100 sieve. To avoid loss of the fine particles of fiber, it may be necessary to make the transfer from the pan to the No. 100 sieve by several stages of washing, stirring the charge, and quickly pouring upon the sieve the fiber flotations, repeating the elutriation procedure several times. The fiber collected on the No. 100 sieve shall then be examined, and the elutriation shall be repeated if it seems desirable.

(*b*) The sieves (or sieve, as the case may be) and the residue contained therein shall then be dried overnight in an oven maintained at a temperature of 45 C. The sieves, or sieve, shall then be carefully inverted over a piece of white paper, and the residual material transferred to the paper by brushing the bottom of the inverted sieve. The transferred material shall then be examined visually, noting whether the separation of fibers from plaster has been complete. The material shall then be transferred to a weighed platinum crucible and dried to constant weight at a temperature of 45 C. If the previous visual examination of the charge on the white paper showed that the fiber was practically free of particles of plaster, the weight of the fiber dried at 45 C., divided by 100, shall be reported as the percentage of fiber. If, on the other hand, the visual examination revealed the presence of an appreciable quantity of plaster associated with the fiber, the contents of the crucible shall be ignited carefully to constant weight. The loss on ignition, divided by 100, shall, in this case, be reported as the percentage of fiber.

COMPRESSIVE STRENGTH OF GYPSUM PARTITION TILE OR BLOCK

Test Specimens

39. Not less than ten full-size tile or block specimens shall be tested. The specimens shall be dried at a temperature of not less than 70 F. (21.1 C.) nor more than 100 F. (37.8 C.) in an atmosphere having a relative humidity of not more than 50 per cent. The specimens shall be weighed at 1-day intervals until constant weight is attained. At the option of the manufacturer or purchaser, strength tests may be conducted on specimens that are dried at room temperature. However, in cases of controversy all test specimens shall be dried to constant weight as herein prescribed.

Procedure

40. (*a*) The specimen shall be tested in the position in which the tile or block is designed to be used, and shall be bedded on and capped with a felt pad not less than $\frac{1}{8}$ nor more than $\frac{1}{4}$ in. in thickness. At the option of the manufacturer or purchaser, or in cases of controversy, the specimens may be suitably capped with calcined gypsum mortar, or the bearing surfaces of the tile may be planed or rubbed smooth and true. When calcined gypsum is used for capping, the test may be conducted after the capping has set and the specimen has been dried to constant weight in accordance with Section 39.

(*b*) The loading head shall completely cover the bearing area of the specimen, and the applied load shall be transmitted through a spherical bearing block of proper design. The speed of the moving head of the testing machine shall be not more than 0.05 in. per min.

FLEXURAL STRENGTH OF GYPSUM BOARDS

Test Specimens

41. Test specimens shall be taken from not less than ten gypsum boards. The test specimens shall be 12 in. in width and a proximately 16 in. in length. The specimens shall be weighed to within 0.5 g. and then stored at a temperature of 60 to 85 F. (15.6 to 29.4 C.) in an atmosphere having a relative humidity of 25 to 50 per cent. The specimens shall be weighed once a day until the weight has become constant to within 0.1 per cent.

Procedure

42. The test specimen shall be centrally supported on fixed parallel bearings spaced 14 in. between centers, and the load-applying block shall be brought in contact with the upper surface of the specimen midway between the supports. All bearings and load surfaces shall be true, shall engage the full width of the test specimen, and shall be rounded to a radius of $\frac{1}{8}$ in. The test load shall be applied at a uniform rate of 60 lb. per min. with a permissible variation of plus or minus 10 per cent in the rate.

Report

43. The results of the flexural strength tests shall be reported for the load applied across the fiber of the surfacing and also parallel to the fiber of the surfacing.

Standard Definitions of

TERMS RELATING TO GYPSUM[1]

A.S.T.M. Designation: C 11 – 41

ADOPTED, 1926; REVISED, 1928, 1939, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 11; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Accelerator.—In the case of stucco, plaster, mortar, concrete, etc., a substance which will hasten the set.

Binder.—In the case of plaster and stucco, etc., a fibrous material, which will increase their cohesiveness, while they are in a plastic state.

Cement.—A material or a mixture of materials (without aggregate) which, when in a plastic state, possesses adhesive and cohesive properties, and which will harden in place.

NOTE.—The word "cement" is used without regard to the composition of the material.

Calcined Gypsum.—Gypsum partially dehydrated by means of heat, having the approximate chemical formula $CaSO_4 \cdot \frac{1}{2}H_2O$.

Crushed Gypsum.—Gypsum subjected to a primary crushing operation.

Gypsum.—Calcium sulfate, combined with two molecules of water, in crystalline form, having the approximate chemical formula $CaSO_4 \cdot 2H_2O$.

Gypsum Lath.—A sheet or slab having an incombustible core of gypsum, containing not more than 15 per cent by weight of fiber, and surfaced with paper suitable to receive gypsum plaster.

Gypsum Molding Plaster.—A material consisting essentially of calcined gypsum for use in making interior embellishments, cornices, etc.

Gypsum Pottery Plaster.—A material consisting essentially of calcined gypsum for use in making pottery molds.

Gypsum Partition Tile or Block.—A gypsum building unit in form of tile or block for use in nonbearing construction in the interior of buildings and for the protection of columns, elevator shafts, etc., against fire.

NOTE 1.—The words "tile" and "block" are synonymous and are used so as to be in accord with certain freight association nomenclature.

NOTE 2.—Gypsum cored tile, 3 or 4 in. in thickness, may be split and used for furring purposes.

Gypsum Sheathing Board.—A sheet or slab having an incombustible core of gypsum, containing not more than 15 per cent by weight of fiber, and surfaced with a water-resistive paper.

Gypsum Wall Board.—A sheet or slab having an incombustible core of gypsum, containing not more than 15 per cent by

[1] Under the standardization procedure of the Society, these definitions are under the jurisdiction of the A.S.T.M. Committee C-11 on Gypsum.

[2] Prior to adoption as standard, these definitions were published as tentative from 1916 to 1926, being revised in 1921, 1922, 1924, and 1925.

A "tentative revision" of this standard appears in the Tentative Revisions of Standards section at the back of this book.

weight of fiber, and surfaced with paper suitable to receive decoration.

Keene's Cement.—Anhydrous calcined gypsum, the set of which is accelerated by the addition of other materials.

Lath.—A material whose primary function is that of a base or background for the reception of plaster or stucco.

Mortar.—A material used in a plastic state, which can be troweled, and becomes hard in place, to bond units of masonry structures.

NOTE.—The word "mortar" is used without regard to the composition of the material, and is defined only with reference to its use as a bonding material as contrasted with the words "stucco" and "plaster."

Plaster.—A material used in a plastic state, which can be troweled, to form a hard covering for the interior surfaces, walls, ceilings, etc., in any building or structure.

NOTE 1.—The word "plaster" is used without regard to the composition of the material, and is defined only with reference to its use and location of use as contrasted with the words "stucco" and "mortar."

NOTE 2.—This does not include decorative media ordinarily applied with a brush.

Retarder.—In the case of stucco, plaster, mortar, concrete, etc., a substance which will retard the set.

Sized Gypsum.—Crushed gypsum of prescribed size of individual particles.

Stucco.—A material used in a plastic state, which can be troweled, to form a hard covering for the exterior walls or other exterior surfaces of any building or structure.

NOTE.—The word "stucco" is used without regard to the composition of the material, and is defined only with reference to its use and location of use as contrasted with the words "plaster" and "mortar."

Wood Fiber.—A material produced by grinding or shredding wood.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: A75.1-1942

Standard Specifications for

CONCRETE BUILDING BRICK[1]

A.S.T.M. Designation: C 55 - 37

ADOPTED, 1934; REVISED, 1937.[2]

Reapproved in 1944 Without Change.

This Standard of the American Society for Testing Materials is issued under the fixed designation C 55; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. (*a*) These specifications cover concrete building brick manufactured from a mixture of portland cement and suitable aggregates such as sand, gravel, crushed stone, bituminous or anthracite cinders, burned clay or shale, or blast-furnace slag, and intended for use in brick masonry. Two grades[3] of brick are covered:

Grade A.—Brick intended for use where exposed to temperature below freezing in the presence of moisture.

Grade B.—Brick intended for use as back-up or interior masonry.

NOTE.—As an example, brick in exposed masonry protected by a facing of 3 in. or more of suitable masonry.

(*b*) When brick are required having strengths greater than prescribed by these specifications, the purchaser should specify the desired minimum compressive strength.

(*c*) If brick having a particular color, texture, finish, or uniformity are desired, these features should be specified separately by the purchaser.

Physical Properties

2. (*a*) The brick shall conform to the physical requirements for the grade[3] specified, as prescribed in Table I.

TABLE I.—PHYSICAL REQUIREMENTS.

Designation	Minimum Compressive Strength (brick flatwise), psi., average gross area		Minimum Modulus of Rupture (brick flatwise) psi., average gross area	
	Average of 5 Brick	Individual	Average of 5 Brick	Individual
Grade A.....	2500	2000	450	300
Grade B.....	1250	1000	300	200

(*b*) Unless otherwise specified by the purchaser, brick of grade A shall be accepted in lieu of grade B.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-15 on Manufactured Masonry Units.

[2] Prior to adoption as standard, these specifications were published as tentative from 1924 to 1934, being revised in 1928 and 1933. Editorially revised and rearranged in 1939.

[3] Attention is called to the fact that when these specifications were editorially revised and rearranged in 1939 the designations of grades A and B were interchanged. The present grade A was formerly designated grade B, and grade B was designated grade A in the standard as published in the 1937 Supplement to Book of A.S.T.M. Standards, Part II.

Size

3. Brick shall conform to the following dimensions:

	Dimension, in.	Permissible Variation, in.
Depth.....	$2\frac{1}{4}$	$\frac{1}{16}$
Width.....	$3\frac{3}{4}$	$\frac{1}{8}$
Length.....	8	$\frac{1}{4}$

Visual Inspection

4. Brick shall pass a visual inspection for freedom from cracks and irregularity.

Sampling and Testing

5. (*a*) For purpose of tests, brick that are representative of the commercial product shall be selected by a competent person appointed by the purchaser, the place or places of selection to be designated when the purchase order is placed. The manufacturer or the seller shall furnish specimens for tests without charge.

(*b*) The brick shall be sampled and tested in accordance with the Standard Methods of Sampling and Testing Brick (A.S.T.M. Designation: C 67) of the American Society for Testing Materials.[4]

[4] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

AMERICAN ASSOCIATION STATE
HIGHWAY OFFICIALS STANDARD
A.A.S.H.O. NO.: M 114-41

Standard Specifications for

BUILDING BRICK (MADE FROM CLAY OR SHALE)[1]

A.S.T.M. Designation: C 62 – 44

ADOPTED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 62; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover brick made from clay or shale and burned, and intended for use in brick masonry (see Explanatory Note). Three grades of brick are covered:

Grade SW.—Brick intended for use where a high degree of resistance to frost action is desired and the exposure is such that the brick may be frozen when permeated with water.

NOTE.—As a typical example, brick used for foundation courses and retaining walls in portions of the United States subject to frost action should conform to this grade. Compliance with this grade is also recommended where a high and uniform degree of resistance to disintegration by weathering is desired.

Grade MW.—Brick intended for use where exposed to temperatures below freezing but unlikely to be permeated with water or where a moderate and somewhat nonuniform degree of resistance to frost action is permissible.

NOTE.—As a typical example, brick used in the face of a wall above ground should conform to this grade. Such exposure is not likely to result in permeation of a brick by water if horizontal surfaces are protected. (See discussion of grade MW in Explanatory Note.)

Grade NW.—Brick intended for use as back-up or interior masonry, or if exposed, for use where no frost action occurs; or if frost action occurs where the average annual precipitation is less than 20 in.

Physical Properties

2. (*a*) *Durability.*—The brick shall conform to the physical requirements for the grade specified, as prescribed in Table I.

(*b*) Unless otherwise specified by the purchaser, brick of grades *SW* and *MW* shall be accepted in lieu of grade *NW*, and grade *SW* in lieu of grade *MW*.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-15 on Manufactured Masonry Units.

[2] Prior to adoption as standard, these specifications were published as tentative from 1927 to 1929, being revised in 1928. They were adopted in 1929, published as standard from 1929 to 1938, being revised in 1930. A revision in the form of separate tentative specifications was issued from 1935 to 1944, being revised in 1936, 1937, 1939, 1940, and 1941.

Prior to their publication as tentative in 1927, these specifications comprised a portion of the Standard Specifications for Building Brick (C 21 - 20) which were published as tentative from 1919 to 1920, being revised in 1920, adopted in 1920, but withdrawn and replaced by C 62 - 27 T in 1927.

(*c*) If the average compressive strength is greater than 8000 psi. *or* the average water absorption is less than 8.0 per cent after 24-hr. submersion in cold water, the requirement for saturation coefficient shall be waived.

(*d*) *Freezing and Thawing.*—The requirements specified in Paragraph (*a*) for water absorption (5-hr. boiling) and saturation coefficient shall be waived provided a sample of five brick, meeting all other requirements, complies with the following requirements when subjected to 50 cycles of the freezing-and-thawing test:[a]

Grade SW..... No breakage and not greater than 1.0 per cent loss in dry weight of any individual brick.

Grade MW..... No breakage and not greater than 3.0 per cent loss in dry weight of any individual brick.

[a] Brick are not required to conform to the provisions of Section 2 (*d*), and these do not apply unless the sample fails to conform to the requirements for absorption and saturation coefficient prescribed in Table I as well as the strength and absorption requirements in Section 2 (*c*).

A particular lot or shipment shall be given the same grading as a previously tested lot, without repeating the freezing-and-thawing test, provided the brick are made by the same manufacturer from similar raw materials and by the same method of forming; and provided also that a sample of five brick selected from the particular lot has an average and individual minimum strength not less than a previously graded sample, and has average and individual maximum water absorption and saturation coefficient not greater than those of the previously tested sample graded according to the freezing-and-thawing test.

(*e*) *Strength.*—When brick are required having strengths greater than prescribed by these specifications, the purchaser should specify the desired minimum compressive strength.

NOTE.—A recommended classification is that given in "Recommended Minimum Requirements for Masonry Wall Construction," Report of Building Code Committee of U. S. Department of Commerce (1931). In this report the following four grades of brick according to compressive strength, tested flatwise, are recognized: 1500 to 2500 psi, 2500 to 4500 psi, 4500 to 8000 psi, and over 8000 psi.

TABLE I.—PHYSICAL REQUIREMENTS.

Designation	Minimum Compressive Strength (brick flatwise), psi., gross area		Maximum Water Absorption by 5-hr. Boiling, per cent		Maximum Saturation Coefficient[a]	
	Average of 5 Brick	Individual	Average of 5 Brick	Individual	Average of 5 Brick	Individual
Grade SW.....	3000	2500	17.0	20.0	0.78	0.80
Grade MW.....	2500	2200	22.0	25.0	0.88	0.90
Grade NW.....	1500	1250	no limit	no limit	no limit	no limit

[a] The saturation coefficient is the ratio of absorption by 24-hr. submersion in cold water to that after 5-hr. submersion in boiling water.

(*f*) *Appearance.*—If brick having a particular color, texture, finish, uniformity or freedom from cracks, warpage, exposed stones, pebbles, or particles of lime are desired, these features should be specified separately by the purchaser, or a sample or samples should be selected to serve as a standard of comparison to which the brick furnished shall conform.

NOTE.—Minor identations or surface cracks, incidental to the usual method of manufacture or the small chipping resulting from the customary methods of handling in shipment and delivery should not be deemed grounds for rejection unless particularly specified to the contrary.

Size and Coring

3. (*a*) *Size.*—The size of brick shall be as specified by the purchaser within permissible variations of plus or minus $\frac{1}{16}$ in. in depth, plus or minus $\frac{1}{8}$ in. in width, and plus or minus $\frac{1}{4}$ in. in length.

NOTE.—The standard size of brick is $2\frac{1}{4}$ by $3\frac{3}{4}$ by 8 in. At present, brick of this size are not produced in some parts of the United States and purchasers should ascertain the size or sizes available.

(*b*) *Coring.*—The net cross-sectional area of brick shall be at least 75 per cent of the gross cross-sectional area (including holes), when measured in a plane parallel with the bearing surface of the brick.

Visual Inspection

4. (*a*) The brick, as delivered to the site, shall, by visual inspection, conform to the requirements specified by the purchaser or to the sample or samples approved as the standard of comparison and to the samples passing the tests for physical requirements.

(*b*) Unless otherwise agreed upon by the purchaser and the seller, a delivery of brick shall contain not more than 5 per cent of broken brick (bats).

Sampling and Testing

5. (*a*) For purpose of tests, brick that are representative of the commercial product shall be selected by a competent person appointed by the purchaser, the place or places of selection to be designated when the purchase order is placed. The manufacturer or the seller shall furnish specimens for tests without charge.

(*b*) The brick shall be sampled and tested in accordance with the Standard Methods of Sampling and Testing Brick (A.S.T.M. Designation: C 67) of the American Society for Testing Materials.[3]

Cost of Tests

6. Unless otherwise specified in the purchase order, the cost of tests shall be borne as follows:

(*a*) If the results of the tests show that the brick do not conform to the requirements of these specifications, the costs shall be borne by the seller.

(*b*) If the results of the tests show that the brick do conform to the requirements of these specifications, the costs shall be borne by the purchaser.

EXPLANATORY NOTE

Extensive tests of brick masonry as well as observations of masonry structures in the field indicate that the important properties of brick which affect the appearance and performance of masonry in buildings are size, color and texture, compressive strength, durability, and suction when laid. Data indicate that a low rate of suction (20 g. per min. or less) is desirable both from the standpoint of bond and watertightness, and since the suction rate of brick that normally have high rates of suction can be reduced to any predetermined value by wetting before laying, this property should not be included in specifications for brick, but may properly be made a part of specifications governing workmanship.

Other properties of brick such as density, soluble salt content, and homogeneity probably also affect the performance of the masonry. Data are not available, however, from which measures of those properties or their effects upon the masonry may be determined, and consequently there are no bases for controlling these factors through a specification. Their effects may be best judged by the record of performance of similar products.

These specifications provide a basis for specifying the following properties of brick, which, as indicated above, appear to be important:

Size.—The dimensions of the standard size brick, together with permissible variations, are given in Section 3 of these specifications. Many brick and solid units are produced in other

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

sizes, however, and when sizes other than standard are acceptable or required, such sizes, together with permissible variations in dimensions, should be specified by the purchaser.

Color and Texture.—Brick are manufactured in a wide variety of colors and textures, neither of which have been standardized. Both color and texture are difficult to describe and a complete list of the products now produced, if obtainable, would be too voluminous to include in a specification. These properties are covered in Section 2 (*f*) of these specifications, which provides that color, texture, finish, and uniformity should be specified by the purchaser. The common practice is to refer to an approved sample.

Compressive Strength.—The compressive strength of brick produced in the United States ranges from 1000 psi. or less (under-burned) to over 20,000 psi. Data are available from which the compressive strength of masonry walls may be predicted with reasonable accuracy if the strength of the brick and the strength of the mortar are known. In the great majority of cases, however, the compressive stresses in masonry walls are relatively low (under 100 psi.) and for such structures minimum compressive strengths of brick of from 1500 to 2500 psi. are ample. These minimum values are included in Section 2 (*a*) of these specifications; when brick having higher strengths are desired, the required strength should be specified by the purchaser as provided in Section 2 (*e*). Strength gradings as set up by the Building Code Committee of the U. S. Department of Commerce are included in this section.

Durability.—Experience has indicated that any well-burned brick will resist the action of freezing and thawing over a long period of time and, from a structural standpoint, may be considered durable. There is a reasonably close correlation between the performance of brick in the freezing-and-thawing test and under the agents of weathering in masonry structures, and at the present time this test appears to be the best measure of the durability of brick. Freezing-and-thawing tests consist of subjecting the brick to alternate cycles (50 or more) of freezing and thawing in the presence of moisture, which requires a period of 10 weeks or more to complete. This makes it impractical as an acceptance test, and for this reason extensive research has been carried on to correlate other physical properties of brick with their resistance to the freezing-and-thawing test.

For brick produced of the same raw material and by the same method of manufacture, either compressive strength or total absorption may be taken as fairly accurate measures of the resistance of such brick to the freezing-and-thawing test; however, limits on these properties that apply to one product do not apply to products produced from different raw materials or by different manufacturing processes, and consequently they alone cannot be used as measures of durability in general specifications. A third property known as "saturation coefficient", when used in conjunction with compressive strength and total absorption by 5-hr. boiling, has been found to provide a means of predicting the resistance of most types of brick to freezing-and-thawing tests with greater accuracy than any other method developed to date.

The saturation coefficient is the ratio of the absorption by 24-hr. submersion in cold water to the absorption after 5-hr. submersion in boiling water and is defined generally as the ratio of easily filled to total fillable pore space. The theory of the saturation coefficient is that if only a part of the total pore space is occupied by water there is room for expansion on freezing into the remaining pore space without disruption of the material. The data indicate that if the easily fillable pore space, that is, the maximum water that might be absorbed by a brick in a wall subjected to excessive moisture does not exceed 80 per cent of the total pore space, the remaining space will relieve the pressure due to expansion on freezing.

While this theory seems to be applicable to many types of brick, it has been found that it does not apply to certain types of de-aired products. Strength and absorption are, therefore, used as measures of durability for these products and their acceptance should be based upon Section 2 (*c*) of these specifications.

The relationship also does not appear to hold for some brick of very high absorption (exceeding the maximum permitted in these specifications) and the acceptance of these products should be in accordance with Section 2 (*d*) which provides for special measures based upon actual freezing-and-thawing tests of the particular product.

In classifying brick according to their resistance to the freezing-and-thawing test, they fell into three general groups as indicated in Table I of these Specifications:

Grade SW which are not affected by the test, and whose appearance and structure remain unchanged.

Grade MW which are for the most part well-burned brick but may include some brick which change materially in appearance when exposed to weathering.

The limits for absorption and saturation coefficient in the grade MW classification have been set to include the average production of

those districts which do not grade or classify their kiln output beyond elimination of extremely underburned (salmon) brick. These "kiln run" shipments frequently include a small percentage of brick which, on exposure to weathering, will lose their surfaces by powdering, flaking, or spalling, and thus produce an unsightly appearance of the exposed masonry surface. Data indicate that brick cannot be classified into intermediate durability. Actually grade MW includes a mixture of durable and nondurable brick. It should be emphasized, however, that disintegration is not necessarily a characteristic of brick in this grade. Certain plants may supply brick under the grading, all of which remain unchanged in appearance even under severe conditions of exposure. The purchaser is advised to examine the field behavior of brick in districts where production classifies as grade MW and reach his own decision as to whether the appearance and condition of masonry at the age of 10 or 20 years is satisfactory.

Grade NW which includes underburned brick that will disintegrate when subjected to freezing and thawing. Such brick should not be used in structures that will be subjected to severe weathering.

In using these specifications the purchaser is urged to consider both the requirements of the structure and the physical properties of the brick available. To a degree at least, brick are a natural product, since such properties as color, compressive strength, and absorption are more or less inherent in the raw material and frequently can be changed only within narrow limits by different methods of manufacture. While the committee believes that the specifications as they now stand provide the best means available of specifying the desirable properties of brick, it recognizes that the specifications are not perfect and that due to the wide variation in raw materials and methods of manufacture, it is probable that some brick which do not conform to the requirements of grade SW still have satisfactory durability. It may also be true that some products which meet these requirements, particularly of grade MW, do not have satisfactory resistance to weathering. For this reason and because of the lack of data on some properties that may have an important bearing upon the performance of masonry, the purchaser should be guided to a degree by the record of performance of any particular product.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: A78.1-1942

Standard Specifications for
SAND-LIME BUILDING BRICK[1]

A.S.T.M. Designation: C 73 – 39

ADOPTED, 1939.[2]

Reapproved in 1944 Without Change.

This Standard of the American Society for Testing Materials is issued under the fixed designation C 73; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. (*a*) These specifications cover brick made from sand and lime and intended for use in brick masonry. Three grades of brick are covered:

Grade SW.—Brick intended for use where exposed to temperature below freezing in the presence of moisture.

NOTE.—As a typical example, brick used for foundation courses and parapets in the northeastern quarter of the United States should conform to grade SW.

Grade MW.—Brick intended for use where exposed to temperature below freezing but unlikely to be saturated with water.

NOTE.—As a typical example, brick exposed in the face of the wall other than parapet or foundations, or brick intended for structures located in regions of the United States characterized by less severe frost action or by drier climate than is found in the northeastern quarter of the United States, should conform to grade MW.

Grade NW.—Brick intended for use as back-up or interior masonry or if exposed, for use where no frost action occurs; or if frost action occurs where the average annual precipitation is less than 15 in.

TABLE I.—PHYSICAL REQUIREMENTS.

Designation	Minimum Compressive Strength (brick flatwise), psi., average gross area		Minimum Modulus of Rupture (brick flatwise), psi., average gross area	
	Average of 5 Brick	Individual	Average of 5 Brick	Individual
Grade SW.....	4500	3500	600	400
Grade MW.....	2500	2000	450	300
Grade NW.....	1500	1500	300	200

(*b*) When brick are required having strengths greater than prescribed by these specifications the purchaser should specify the desired minimum compressive strength.

(*c*) If brick having a particular color, texture, finish, or uniformity are desired,

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-15 on Manufactured Masonry Units.

[2] Prior to their present adoption as standard, these specifications were published as tentative from 1928 to 1930. They were adopted in 1930, published as standard from 1930 to 1938, being revised in 1937, but withdrawn and republished as tentative from 1938 to 1939.

these features should be specified separately by the purchaser.

Physical Properties

2. (*a*) The brick shall conform to the physical requirements for the grade specified, as prescribed in Table I.

(*b*) Unless otherwise specified by the purchaser, brick of grades SW and MW shall be accepted in lieu of grade NW and grade SW in lieu of grade MW.

Size

3. Brick shall conform to the following dimensions:

	Dimension, in.	Permissible Variation, plus or minus, in.
Depth	$2\frac{1}{4}$	$\frac{1}{8}$
Width	$3\frac{3}{4}$	$\frac{1}{8}$
Length	8	$\frac{1}{4}$

Visual Inspection

4. Brick shall pass a visual inspection for soundness, compact structure, reasonably uniform shape, and freedom from the following: cracks, warpage, large pebbles, balls of clay, or particles of lime that would affect the serviceability or strength of the brick.

Sampling and Testing

5. (*a*) For purpose of tests, brick that are representative of the commercial product shall be selected by a competent person appointed by the purchaser, the place or places of selection to be designated when the purchase order is placed. The manufacturer or the seller shall furnish specimens for tests without charge.

(*b*) The brick shall be sampled and tested in accordance with the Standard Methods of Sampling and Testing Brick (A.S.T.M. Designation: C 67) of the American Society for Testing Materials.[3]

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for

PAVING BRICK[1]

A.S.T.M. Designation: C 7 - 42

ADOPTED, 1942.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 7; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover vitrified clay brick for use in the construction of pavements.

Manufacture

2. Paving brick shall be manufactured from fire clay, semi-fire clay, or shale, or combinations thereof. The manufacturing process shall be such that the brick are thoroughly annealed, tough, durable, and evenly burned so that, when broken, they show a uniformly dense structure free from lime, air pockets and marked laminations. The name of the brick or the manufacturer shall be impressed on the side or end of each brick in recessed letters.

Lugs

3. (*a*) *Side Lugs.*—The brick shall be provided on one side with not less than two lugs, which shall be $\frac{3}{16}$ in. in height with a permissible variation of plus or minus $\frac{1}{16}$ in. The area of the projections or lugs measured at the base of the lug shall not exceed in total area 3 sq. in. No lug shall be continuous from top to bottom of the brick, but shall be of a type that allows free horizontal flow of the filler.

(*b*) *End Lugs.*—Vertical fiber brick shall be provided with not less than two noncontinuous lugs or portions of lugs on each end of each brick. Lugs shall be nonmeshing and shall have a total base area of not more than $2\frac{1}{2}$ sq. in. and shall be manufactured so there may be free horizontal flow of the filler. The lugs shall project from the ends so as to insure a joint opening of $\frac{3}{16}$ in. with a permissible variation of plus or minus $\frac{1}{16}$ in.

Types and Sizes

4. (*a*) Paving brick shall conform to the following types and nominal sizes. The dimensions of the brick shall be construed as applying to the body of the brick only, exclusive of lugs.

	Depth, in.	Width, in.	Length, in.
Repressed lug brick	4	$3\frac{1}{2}$	$8\frac{1}{2}$
Vertical fiber lug brick	$2\frac{1}{2}$	4	$8\frac{1}{2}$
	3	4	$8\frac{1}{2}$
	$3\frac{1}{2}$	4	$8\frac{1}{2}$
Wire-cut lug brick	4	$3\frac{1}{2}$	$8\frac{1}{2}$

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-15 on Manufactured Masonry Units.

[2] Prior to their present adoption as standard, these specifications were published as tentative from 1913 to 1915, being revised in 1914. They were adopted in 1915, published as standard from 1915 to 1937, being revised in 1923 and 1930, but withdrawn, revised, and republished as tentative from 1937 to 1942, being revised in 1938 and 1940.

NOTE.—These sizes and varieties correspond to those described in Simplified Practice Recommendation R 1-40[3] of the U. S. Department of Commerce, and are subject to change from year to year.

(*b*) The edges of repressed brick shall be rounded to a radius of not more than $\frac{3}{16}$ in.

Permissible Variations in Size

5. For any lot of paving brick furnished under these specifications, the individual brick shall not vary from the nominal size requirements specified in Section 4 (*a*) by more than plus or minus $\frac{1}{8}$ in. in either transverse dimension, exclusive of kiln marks, or by more than plus or minus $\frac{1}{4}$ in. in length.

Loss in Rattler Test

6. (*a*) The percentage of loss in the rattler test of the respective sizes of paving brick specified shall not exceed the following limits:

Size of Brick, Transverse Dimensions, in.	Length, in.	Maximum Loss in Rattler Test, per cent
2½ by 4	8½	26
3 by 4	8½	24
3½ by 4	8½	22

(*b*) The total number of pieces weighing 1 lb. or more remaining after the completion of the rattler test shall not exceed 12.

Visual Inspection

7. Paving brick shall be subject to visual inspection prior to shipment, after delivery, and during laying for the purpose of culling out and rejecting individual imperfect brick. Subject to the exceptions specified in Paragraph (*b*), individual brick may be culled and rejected for the following causes:

(*a*) *Variation in Size.*—Brick which vary from the nominal size specified in Section 4 (*a*) by more than plus or minus $\frac{1}{8}$ in. in either transverse dimension, exclusive of kiln marks, or by more than plus or minus $\frac{1}{4}$ in. in length.

(*b*) *Broken or Chipped Brick.*—Brick which are chipped or broken so that neither wearing surface remains substantially intact, or so that the lower, or bearing surface is reduced in area more than 15 per cent, or are weakened structurally. However, brick so rejected but otherwise acceptable may be used for part-brick for beginning courses and for batting provided the portion so used conforms to all of the requirements for visual inspection.

(*c*) *Cracked Brick.*—Brick which are cracked to such a degree, either from shocks received in shipping and handling or from defective manufacture, as to produce defects described in Paragraph (*b*), unless such cracks are plainly superficial, and insufficient to weaken the brick under the conditions of use.

(*d*) *Warped or Kiln-Marked Brick.*—Brick which are kiln-marked more than $\frac{3}{16}$ in. or which are misshapen, bent, or twisted so that they will not form a proper surface, or align with other brick in conformance with the specifications for laying brick in the pavement. Vertical fiber brick shall have no kiln-marks in the wire cut surface.

(*e*) *Under-Burned Brick.*—Brick which are obviously too soft and too poorly vitrified to endure traffic wear. If disagreement on this requirement arises between the purchaser and the seller it shall be the right of the purchaser to make one or more rattler tests of the brick he wishes to exclude, and if in any or all tests the brick fail to conform to the requirements of these specifications, then all brick having the same objectionable appearance may be excluded.

Basis of Acceptance

8. Acceptance of any lot of paving brick shall be based on the results of

[3] Reaffirmed without change June 29, 1943. Copies are available at 5 cents each from the Superintendent of Documents, Government Printing Office, Washington, D. C.

tests on samples representing that lot, subject only to rejection at any time of individually imperfect or unsatisfactory brick, in accordance with the requirements under Section 6.

Retesting

9. (*a*) If the percentage of loss in the rattler test on any sample exceeds by not more than 2 per cent the maximum permissible rattler loss specified in Section 6 (*a*) for the size specified, or if the number of pieces weighing 1 lb. or more remaining after the completion of the rattler test exceeds 12 as specified in Section 6 (*b*), two additional tests shall be made. Each of these additional tests shall comply with the requirements of both Section 6 (*a*) and (*b*) and, in addition, the average rattler loss for the three tests shall not exceed the maximum permissible loss specified in Section 6 (*a*).

(*b*) Where a lot of paving brick is rejected for failure to meet the test requirements prescribed in these specifications, the purchaser may, at his option, permit the seller to regrade the rejected brick and again offer the brick for test and acceptance. The regraded brick shall be resampled and tested and if they again fail, the purchaser may definitely and finally reject the lot represented by the test.

Payment for Cost of Testing

10. Unless otherwise agreed, the seller shall furnish at the place of manufacture, without charge to the purchaser, the use of an approved rattler in efficient operating condition, conforming to the requirements specified in Section 12. Payment of the cost of testing shall be made by the purchaser, except that the cost of testing additional samples in accordance with Section 9, made necessary by the failure of any lot or lots, or of testing supplemental samples to determine the acceptability of a portion of any lot or lots, shall be paid by the seller; provided, however, that if the supplemental tests show the brick under examination to be acceptable, then the cost of such tests shall be paid by the purchaser.

RATTLER TEST

Sampling

11. (*a*) *Selection of Sample.*—The sample for the rattler test shall consist of ten whole undamaged brick which conform individually to the visual inspection requirements specified in Section 7. Samples shall preferably be selected at the place of manufacture and tested prior to shipment. However, in special cases where less than 100,000 brick are involved, samples, when required, may be selected subsequent to delivery at the destination.

(*b*) *Number of Samples.*—In general, one sample shall be selected for each 15,000 brick or fraction thereof; provided, however, that when the sampling is done at the place of manufacture prior to shipment and the total number of brick involved is more than 100,000 brick, the number of brick for which one sample is considered representative may be increased at the option of the purchaser to not more than 50,000 brick.

Apparatus

12. The machine[4] used in the rattler test shall be of good mechanical construction, self-contained, and shall conform to the following details as regards the material and dimensions. It shall consist of a barrel, frame, and driving mechanism as described in Sections 13 to 15.

[4] A detailed illustration of a rattler showing the construction of a machine which will meet the requirements specified is available from the Headquarters of the American Society for Testing Materials, 260 S. Broad St., Philadelphia 2, Pa. This illustration was published previously as Plate I of the 1936 Book of A.S.T.M. Standards, Part II, p. 128.

Barrel

13. The barrel of the machine shall consist of the heads, headliners, staves, and staveliners comforming to the following requirements:

(*a*) *Heads.*—The heads may be cast in one piece with the trunnions which shall be $2\frac{7}{16}$ in. in diameter and shall have a bearing 6 in. in length; or they may be cast with heavy hubs which shall be bored out for $2\frac{7}{16}$-in. shafts and shall be key-seated for two keys, each $\frac{1}{2}$ by $\frac{3}{8}$ in. and spaced 90 deg. apart. The shaft shall be a snug fit and when keyed shall be entirely free from lost motion. The distance from the end of the shaft or trunnion to the inside face of the head shall be $15\frac{3}{8}$ in. for the head of the driving end of the rattler and $11\frac{3}{8}$ in. for the other head, and the distance from the face of the hubs to the inside face of the heads shall be $5\frac{1}{8}$ in.

The heads shall not be less than $\frac{3}{4}$ nor more than $\frac{7}{8}$ in. in thickness. In outline, each head shall be a regular 14-sided polygon inscribed in a circle $28\frac{5}{8}$ in. in diameter. Each head shall be provided with flanges not less than $\frac{3}{4}$ in. in thickness, and extending outward $2\frac{1}{2}$ in. from the inside face of the head to afford a means of fastening the staves. The surface of the flanges of the head shall be smooth and give a true and uniform bearing for the staves. To secure the desired true and uniform bearing, the surfaces of the flanges of the head shall be either ground or machined. The flanges shall be slotted on the outer edge, so as to provide for two $\frac{3}{4}$-in. bolts at each end of each stave, the slots to be $1\frac{3}{16}$ in. in width and $2\frac{3}{4}$ in. center to center. Each slot shall be provided with a recess for the bolt head, which shall act to prevent the turning of the same. Between each two slots there shall be a brace $\frac{3}{8}$ in. in thickness extending not less than 2 in. down the outward side of the head.

(*b*) *Headliners.*—There shall be for each head a cast-iron headliner, 1 in. in thickness and conforming to the outline of the head, but inscribed in a circle $28\frac{1}{8}$ in. in diameter. This headliner shall be fastened to the head by seven $\frac{5}{8}$-in. cap screws, through the head from the outside. Whenever these headliners become worn down $\frac{1}{2}$ in. below their initial surface level, at any point of their surface, they shall be replaced with new ones. The metal of these headliners shall be hard machinery iron and should contain not less than 1 per cent of combined carbon.

(*c*) *Staves.*—The staves shall be made of 6-in. medium-carbon steel structural channels $27\frac{1}{4}$ in. in length and weighing 15.5 lb. per lineal foot. The staves shall have two holes $\frac{13}{16}$ in. in diameter drilled in each end, the center line of the holes being 1 in. from the end and $1\frac{3}{8}$ in. either way from the longitudinal center line. The spaces between the staves shall be as uniform as practicable, but shall not exceed $\frac{6}{16}$ in.

(*d*) *Staveliners.*—The interior or flat side of each stave shall be protected by a liner $\frac{3}{8}$ in. in thickness by $5\frac{1}{2}$ in. in width by $19\frac{3}{4}$ in. in length. The liner shall consist of medium-carbon steel plate, and shall be riveted to the channel by three $\frac{1}{2}$-in. rivets, one of which shall be on the center line both ways and the other two on the longitudinal center line and spaced 7 in. from the center each way. The rivet holes shall be countersunk on the face of the liner and the rivets shall be driven hot and chipped off flush with the surface of the liners. These liners shall be inspected from time to time, and if found loose shall be at once re-riveted.

(*e*) *Relining.*—No stave shall be used for more than 70 consecutive tests without renewing its lining. Two of the 14 staves shall be removed and relined at a time in such a way that of each

pair one falls upon one side of the barrel and the other upon the opposite side, and also so that the staves changed shall be consecutive but not contiguous; for example, 1 and 8, 3 and 10, 5 and 12, 7 and 14, 2 and 9, 4 and 11, 6 and 13, etc., to the end that the interior of the barrel at all times shall present the same relative conditions of repair. The changes in the staves should be made at the time when the shot charges are being corrected, and the record must show the number of charges run since the last pair of new lined staves was placed in position.

(*f*) *Construction.*—The staves when bolted to the heads shall form a barrel 20 in. in length, inside measurement, between headliners. The liners of the staves shall be so placed as to drop between the headliners. The staves shall be bolted tightly to the heads by four $\frac{3}{4}$-in. bolts and each bolt shall be provided with a locknut and shall be inspected at not less frequent intervals than every fifth test and all nuts kept tight. A record shall be made after each inspection showing in what condition the bolts were found.

Barrel Frame

14. The barrel shall be mounted on a cast-iron frame of sufficient strength and rigidity to support it without undue vibration. It shall rest on a rigid foundation with or without the interposition of wooden plates, and shall be fastened thereto by bolts at not less than four points.

Driving Mechanism

15. The barrel shall be driven by gearing whose ratio of driver to driven is not less than one to four. The counter shaft upon which the driving pinion is mounted shall not be less than $1\frac{15}{16}$ in. in diameter, with bearing not less than 6 in. in length. If a belt drive is used, the pulley shall be not less than 18 in. in diameter and $6\frac{1}{2}$ in. in face. A belt at least 6 in. in width, properly adjusted to avoid unnecessary slipping, should be used.

Preliminary Operation

16. When a new rattler in which a complete set of new staves is furnished is first put into operation, it shall be charged with 400 lb. of shot of the same sizes and in the same proportion as provided in Section 17, and shall then be run for 18,000 revolutions at the rate of speed specified in Section 21 (*a*). The shot shall then be removed and a standard shot charge inserted, after which the rattler may be charged with brick for a test.

Abrasive Charge

17. The abrasive charge shall consist of cast-iron spheres of two sizes:

(*a*) *Large Spheres.*—When new, the larger spheres shall be 3.75 in. in diameter and shall weigh approximately 7.5 lb. (3.40 kg.) each. Ten spheres of this size shall be used. These shall be weighed separately after each ten tests, and if the weight of any large sphere falls to 7 lb. (3.175 kg.) it shall be discarded and a new one substituted; provided, however, that all of the large spheres shall not be discarded and substituted by new ones at any single time, and that in so far as possible the large spheres shall compose a graduated series in various stages of wear.

(*b*) *Small Spheres.*—When new, the smaller spheres shall be 1.875 in. in diameter and shall weigh approximately 0.95 lb. (0.43 kg.) each. In general, the number of small spheres in a charge shall not fall below 245 nor exceed 260. The collective weight of the large and small spheres shall be as nearly 300 lb. as possible. No small sphere shall be retained in use after it has been worn

down so that it will pass a circular hole 1.75 in. in diameter, drilled in an iron plate ¼ in. in thickness, or weigh less than 0.75 lb. (0.34 kg.)

Further, the small spheres shall be tested by passing them over the plate mentioned or by weighing, after every ten tests, and any which pass through or fall below the specified weight shall be replaced by new spheres; provided, however, that all of the small spheres shall not be rejected and replaced by new ones at any one time, and that in so far as possible the small spheres shall compose a graduated series in various stages of wear. At any time that any sphere is found to be broken or defective it shall at once be replaced.

Chemical Composition of Abrasive Charge

18. (*a*) The cast-iron abrasive spheres shall conform to the following requirements as to chemical composition:

Combined carbon, min., per cent	2.50
Graphitic carbon, max., per cent	0.25
Manganese, max., per cent	0.50
Phosphorus, max., per cent	0.25
Sulfur, max., per cent	0.08
Silicon, max., per cent	1.00

(*b*) For each new batch of spheres used, the chemical analysis shall be furnished by the maker or be obtained by the user before introducing the spheres into the abrasive charge, and unless the analysis conforms to the requirements specified the batch of spheres shall be rejected.

Scales

19. The scales used shall be placed on a solid level foundation and shall be sufficiently sensitive to determine the initial weight of 10 brick to the nearest 0.1 lb. The scale shall be tested by a standard test weight at intervals of not more than every ten tests.

Test Specimen

20. The number of brick per test shall be ten whole brick for all weights of brick, and the brick shall be weighed to the nearest 0.1 lb. Brick shall be surface dry when tested. No brick should be selected as part of a regular test that would be rejected by any other requirements of the specifications under which the purchase is made.

Procedure

21. (*a*) The test specimen and the abrasive charge shall be placed in the rattler and the machine rotated for 1800 revolutions at a rate of not less than 29.5 nor more than 30.5 rpm. A counting machine shall be attached to the rattler for counting the revolutions. A margin not to exceed ten revolutions will be allowed for starting and stopping. Only one start and stop per test is, in general, acceptable. If from accidental causes, the rattler is stopped and started more than once during a test, and the percentage loss exceeds the maximum permissible under the specifications, the test shall be disqualified and another made.

(*b*) Any test, at the expiration of which a staveliner is found detached from the stave or seriously out of position, shall be rejected.

Percentage Loss of Weight

22. The loss shall be calculated as a percentage of the original weight of the brick composing the charge, by weighing the brick remaining in the rattler at the end of the test, deducting this weight from the original weight of the brick, dividing this difference by the original weight of the brick and multiplying by 100, as follows:

$$\text{Percentage of loss} = \frac{\text{original wt.} - \text{wt. after testing}}{\text{original wt.}} \times 100$$

In weighing the rattled brick, any piece weighing less than 1 lb. shall be rejected.

Rattler Record

23. (*a*) A complete and continuous record shall be kept of the operation of all rattlers working under the rattler test. This record shall contain the following data concerning each test made:

1. The name of the person, firm, or corporation furnishing each sample tested.
2. The name of the manufacturer of the brick represented in each sample tested.
3. The name of the street or contract which the sample represented.
4. The brands or marks upon the brick by which they were identified.
5. The number of brick furnished.
6. The date on which they were received for test.
7. The date on which they were tested.
8. The drying treatment given before testing, if any.
9. The length, width, and depth of the brick.
10. The collective weight of the ten large spherical shot used in making the test, at the time of their last standardization.
11. The number and collective weight of the small spherical shot used in making the test, at the time of their last standardization.
12. The total weight of the shot charge, after its last standardization.
13. Certificate of the operator that he examined the condition of the machine as to staves, liners, and any other parts affecting the barrel, and found them to conform to the requirements at the beginning of the test.
14. Certificate of the operator as to the number of charges tested since the last standardization of shot charge and last renewals of stave liners.
15. The time of the beginning and ending of each test and the number of revolutions made by the barrel during the test, as shown by the counting machine.
16. Certificate of the operator as to the number of stops and starts made in each test.
17. The initial collective weight of the ten brick composing the charge and their collective weight after rattling.
18. The number of broken brick and remarks upon the portions which were included in the final weighing.
19. General remarks upon the test and any irregularities occurring in its execution.
20. The certificate of the operator that the test was made in accordance with the requirements of the American Society for Testing Materials and that the record is a true record.
21. The signature of the operator or person responsible for the test.
22. The location of the laboratory where the test was made and name of the owner.
23. The serial number of the test.

(*b*) In the event of more than one copy of the record of any test being required, they may be furnished on separate sheets and marked duplicate, but the original record shall always be preserved intact and complete.

Standard Specifications for

SEWER BRICK (MADE FROM CLAY OR SHALE)[1]

A.S.T.M. Designation: C 32 - 42

ADOPTED, 1942.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 32; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover brick made from clay or shale and burned, and intended for use in drainage structures for the conveyance of sewage, industrial wastes, and storm water. Three grades of brick are covered:

Grade SA.—Brick intended for use in structures requiring imperviousness and resistance to the action of sewage carrying large quantities of abrasive material at velocities exceeding 8 ft. per sec.

Grade MA.—Brick intended for use in structures requiring imperviousness and resistance to the action: (*1*) of sewage free from abrasive materials, and (*2*) of sewage carrying abrasive materials at velocities of 8 ft. per sec. or less.

Grade NA.—Brick intended for use in structures not requiring high degrees of imperviousness nor of abrasive resistance.

NOTE.—Brick of grade *NA* are suitable for use in catch basins, arches, the upper portions of manholes, and for backing.

TABLE I.—PHYSICAL REQUIREMENTS.

Designation	Minimum Compressive Strength, (brick flatwise), psi., average gross area		Maximum Water Absorption by 5-hr. Boiling, per cent	
	Average of 5 Brick	Individual	Average of 5 Brick	Individual
Grade *SA*.....	8000	5000	6	9
Grade *MA*[a].....	5000	3000	12	16
Grade *NA*[a].....	2500	2000	20	24

[a] Where resistance to frost action in the presence of moisture is required, grades *MA* and *NA* shall conform to the additional requirement that the saturation coefficient (*C/B*), that is, ratio of absorption by 24-hr. submersion in cold water to that after 5 hr. submersion in boiling water, shall not exceed 0.80.

Physical Properties

2. (*a*) The brick shall conform to the physical requirements for the grade specified, as prescribed in Table I.

(*b*) Unless otherwise specified by the purchaser, brick of grade *SA* shall be accepted in lieu of grade *MA*; also grades *SA* and *MA* shall be accepted in

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-15 on Manufactured Masonry Units.

[2] Prior to their present adoption as standard, these specifications were published as tentative from 1921 to 1923. They were adopted in 1923, published as standard from 1923 to 1937, being revised in 1924, but withdrawn, revised, and republished as tentative from 1937 to 1942, being revised in 1939.

lieu of grade *NA*. Brick having absorption between 12 and 20 per cent shall be graded *MA* if the compressive strength conforms to the requirements for grade *SA*.

Size

3. (*a*) Brick shall be furnished of one or more of the following designated sizes as specified:

Size	Depth, in.	Width, in.	Length, in.
No. 1	$2\frac{1}{4}$	$3\frac{3}{4}$	8
No. 2	$2\frac{1}{2}$	4	$8\frac{1}{2}$
No. 3	3	4	$8\frac{1}{2}$
No. 4	$3\frac{1}{2}$	4	$8\frac{1}{2}$

(*b*) For any lot of sewer brick furnished under these specifications, not more than 2 per cent of the brick shall vary from the nominal size requirements specified in Paragraph (*a*) by more than plus or minus $\frac{1}{8}$ in. in either transverse dimension, or by more than plus or minus $\frac{1}{4}$ in. in length.

Visual Inspection

4. (*a*) Brick shall pass a visual inspection for freedom from the following: cracks, warpage, stones, pebbles, or particles of lime that would affect the serviceability of the brick.

(*b*) Brick shall be of rectangular cross-section with substantially straight, square corners. The ends and at least one edge shall have plain surfaces.

(*c*) When paving brick are used, lugs and grooves will be permitted on one edge and the ends. Kiln marks not exceeding $\frac{3}{16}$ in. in depth shall be permitted on the opposite edge.

NOTE.—Where cored brick are permitted by the purchaser, they shall conform to the requirements of these specifications.

Sampling and Testing

5. (*a*) For purpose of tests, brick that are representative of the commercial product shall be selected by a competent person appointed by the purchaser, the place or places of selection to be designated when the purchase order is placed. The manufacturer or the seller shall furnish specimens for tests without charge.

(*b*) Brick shall be sampled and tested in accordance with the Standard Methods of Sampling and Testing Brick (A.S.T.M. Designation: C 67) of the American Society for Testing Materials.[3]

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for

SEWER BRICK (MADE FROM CLAY OR SHALE)[1]

A.S.T.M. Designation: C 32 - 42

ADOPTED, 1942.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 32; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover brick made from clay or shale and burned, and intended for use in drainage structures for the conveyance of sewage, industrial wastes, and storm water. Three grades of brick are covered:

Grade SA.—Brick intended for use in structures requiring imperviousness and resistance to the action of sewage carrying large quantities of abrasive material at velocities exceeding 8 ft. per sec.

Grade MA.—Brick intended for use in structures requiring imperviousness and resistance to the action: (*1*) of sewage free from abrasive materials, and (*2*) of sewage carrying abrasive materials at velocities of 8 ft. per sec. or less.

Grade NA.—Brick intended for use in structures not requiring high degrees of imperviousness nor of abrasive resistance.

NOTE.—Brick of grade *NA* are suitable for use in catch basins, arches, the upper portions of manholes, and for backing.

TABLE I.—PHYSICAL REQUIREMENTS.

Designation	Minimum Compressive Strength, (brick flatwise), psi., average gross area		Maximum Water Absorption by 5-hr. Boiling, per cent	
	Average of 5 Brick	Individual	Average of 5 Brick	Individual
Grade *SA*.....	8000	5000	6	9
Grade *MA*[a].....	5000	3000	12	16
Grade *NA*[a].....	2500	2000	20	24

[a] Where resistance to frost action in the presence of moisture is required, grades *MA* and *NA* shall conform to the additional requirement that the saturation coefficient (*C/B*), that is, ratio of absorption by 24-hr. submersion in cold water to that after 5 hr. submersion in boiling water, shall not exceed 0.80.

Physical Properties

2. (*a*) The brick shall conform to the physical requirements for the grade specified, as prescribed in Table I.

(*b*) Unless otherwise specified by the purchaser, brick of grade *SA* shall be accepted in lieu of grade *MA*; also grades *SA* and *MA* shall be accepted in

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-15 on Manufactured Masonry Units.

[2] Prior to their present adoption as standard, these specifications were published as tentative from 1921 to 1923. They were adopted in 1923, published as standard from 1923 to 1937, being revised in 1924, but withdrawn, revised, and republished as tentative from 1937 to 1942, being revised in 1939.

lieu of grade *NA*. Brick having absorption between 12 and 20 per cent shall be graded *MA* if the compressive strength conforms to the requirements for grade *SA*.

Size

3. (*a*) Brick shall be furnished of one or more of the following designated sizes as specified:

Size	Depth, in.	Width, in.	Length, in.
No. 1	2½	3¾	8
No. 2	2½	4	8½
No. 3	3	4	8½
No. 4	3½	4	8½

(*b*) For any lot of sewer brick furnished under these specifications, not more than 2 per cent of the brick shall vary from the nominal size requirements specified in Paragraph (*a*) by more than plus or minus ⅛ in. in either transverse dimension, or by more than plus or minus ¼ in. in length.

Visual Inspection

4. (*a*) Brick shall pass a visual inspection for freedom from the following: cracks, warpage, stones, pebbles, or particles of lime that would affect the serviceability of the brick.

(*b*) Brick shall be of rectangular cross-section with substantially straight, square corners. The ends and at least one edge shall have plain surfaces.

(*c*) When paving brick are used, lugs and grooves will be permitted on one edge and the ends. Kiln marks not exceeding $\frac{3}{16}$ in. in depth shall be permitted on the opposite edge.

NOTE.—Where cored brick are permitted by the purchaser, they shall conform to the requirements of these specifications.

Sampling and Testing

5. (*a*) For purpose of tests, brick that are representative of the commercial product shall be selected by a competent person appointed by the purchaser, the place or places of selection to be designated when the purchase order is placed. The manufacturer or the seller shall furnish specimens for tests without charge.

(*b*) Brick shall be sampled and tested in accordance with the Standard Methods of Sampling and Testing Brick (A.S.T.M. Designation: C 67) of the American Society for Testing Materials.[3]

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: A82.1-1944

Standard Methods of

SAMPLING AND TESTING BRICK[1]

A.S.T.M. Designation: C 67 - 44

ADOPTED, 1931; REVISED, 1937, 1939, 1941, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 67; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods cover procedures for the sampling and testing of brick for modulus of rupture (flexure test), compressive strength, absorption, saturation coefficient, effect of freezing and thawing, initial rate of absorption (suction), and efflorescence.

Definitions of Terms

2. The Standard Definitions of Terms Relating to Methods of Testing (A.S.T.M. Designation: E 6) of the American Society for Testing Materials[3] shall be considered as applying to the terms used in these methods.

SAMPLING

Selection of Test Specimens

3. For purpose of tests, full-size brick shall be selected by the purchaser or by his authorized representative. Specimens shall be representative of the whole lot of brick from which they are selected.

Number of Specimens

4. For the modulus of rupture, compressive strength, absorption, and freezing-and-thawing determinations, at least five brick shall be selected from each lot of 50,000 brick or fraction thereof. For lots of more than 500,000 brick, five individual brick shall be selected from each 100,000 brick or fraction thereof contained in the lot. In no case shall less than five brick be taken. Additional specimens may be taken at the discretion of the purchaser.

Identification

5. Each specimen shall be marked so that it may be identified at any time. Markings shall cover not more than 5 per cent of the superficial area of the specimen.

MODULUS OF RUPTURE (FLEXURE TEST)

Test Specimens

6. Five dry full-size brick shall be tested.

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee C-15 on Manufactured Masonry Units.

[2] Prior to adoption as standard, these methods were published as tentative from 1927 to 1931, being revised in 1928 and 1930.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Procedure

7. (*a*) The test specimen shall be supported flatwise on a span of 7 in. and loaded at mid-span. If the specimens have recesses (panels or depressions) they shall be placed so that such recesses are on the compression side. The load shall be applied to the upper surface of the specimen through a steel bearing plate ¼ in. in thickness and 1½ in. in width and of a length at least equal to the width of the specimen. The direction of the load shall be perpendicular to the loaded surface of the specimen.

(*b*) The supports for the test specimen shall be free to rotate in the longitudinal and transverse direction of the test specimen and shall be adjusted so that they will exert no force in these directions.

(*c*) *Speed of Testing.*—The rate of loading shall not exceed 2000 lb. per min., but this requirement may be considered as being met if the speed of the moving head of the testing machine during the application of the load is not more than 0.05 in. per min.

Calculations and Report

8. (*a*) The modulus of rupture of each specimen shall be calculated as follows:

$$\text{Modulus of rupture, psi.} = \frac{3Wl}{2bd^2}$$

where:

W = maximum load indicated by the testing machine, in pounds,
l = distance between the supports in inches (7 in.),
b = average over-all width, face to face, of the specimen, in inches, and
d = average over-all depth, face to face, of the specimen, in inches.

(*b*) The average of the modulus of rupture determinations of all the specimens tested shall be reported as the modulus of rupture of the lot of brick.

COMPRESSIVE STRENGTH

Test Specimens

9. The test specimens shall consist of half brick with approximately plane and parallel ends. A specimen may be obtained from a whole brick by any method that will produce, without shattering, a half brick. Five specimens shall be tested.

NOTE.—This permits the use of suitable half brick available after the flexure test has been made.

Capping Test Specimens

10. (*a*) If the flatwise faces of the test specimen are recessed or paneled, the depressions shall be filled with a neat portland-cement paste, which shall then be aged at least 24 hr. before the specimen is capped.

(*b*) The two opposite flatwise faces of each specimen shall be coated with shellac which shall be allowed to dry thoroughly.

(*c*) One of the dry shellacked faces of the specimen shall be bedded in a thin coat of a neat paste of calcined gypsum (plaster of Paris) that has been spread on an oiled nonabsorbent plate such as glass or a machined metal plate. This procedure shall be repeated with the other dry shellacked face. Care shall be taken that the opposite bearing surfaces so formed will be approximately parallel. The caps shall be aged at least 2 hr. before the specimens are tested.

Procedure

11. (*a*) All specimens shall be tested flatwise (that is, the load shall be applied in the direction of the thickness of the brick).

(*b*) The testing machine shall conform to the requirements of the Standard Methods of Verification of Testing Machines (A.S.T.M. Designation: E 4) of the American Society for Testing Materials.[3]

(*c*) The upper bearing shall be a spherically seated, hardened metal block firmly attached at the center of the upper head of the machine. The center of the sphere shall lie at the center of the surface of the block in contact with the specimen. The block shall be closely held in its spherical seat, but shall be free to turn in any direction. The diameter of the bearing surface shall be at least 5 in. A hardened metal bearing block shall be used beneath the specimen to minimize wear of the lower platen of the machine. The bearing block surfaces intended for contact with the specimen should have a hardness not less than C60 Rockwell number (Brinell number 620). These surfaces shall not depart from plane surfaces by more than 0.001 in. When the bearing area of the spherical bearing block is not sufficient to cover the area of the specimen, a steel plate with surfaces machined to true planes within plus or minus 0.001 in., and with a thickness equal to at least one third of the distance from the edge of the spherical bearing to the most distant corner, shall be placed between the spherical bearing block and the capped specimen.

(*d*) *Speed of Testing.*—When the load on the specimen exceeds approximately one half of the maximum load, the rate of loading shall be such that the load will be read accurately. Preferably, the rate of loading should not exceed 2000 psi. per min., but this requirement may be considered as being met if the speed of the moving head of the testing machine during the application of the load is not more than 0.05 in. per min.

Calculations and Report

12. (*a*) The compressive strength of each specimen shall be calculated as follows:

$$\text{Compressive strength, psi.} = \frac{W}{A}$$

where:

W = maximum load indicated by the testing machine, in pounds, and

A = average of the gross areas of the upper and lower bearing surfaces of the specimen in square inches.

(*b*) The average compressive strength of all the specimens tested shall be reported as the compressive strength of the lot of brick.

ABSORPTION

Accuracy of Weighings

13. The scale or balance used shall have a capacity of not less than 2000 g. and shall be sensitive to 0.5 g.

24-hr. Submersion Test

Test Specimens

14. The test specimens shall consist of half brick conforming to the requirements of Section 9. Five specimens shall be tested.

Procedure

15. (*a*) *Drying.*—The test specimens shall be dried to constant weight in a ventilated oven at 110 to 115 C. (230 to 239 F.). When cool, each specimen shall be weighed.

NOTE 1.—It can be assumed that heating for 72 hr. at these temperatures will assure constant weight except for deaired brick or brick whose absorption is less than 8.0 per cent by 5-hr. submersion in boiling water as determined in accordance with Section 18 (*b*).

NOTE 2.—Storage of brick, unstacked, with separate placement, in a ventilated room for a period of 4 hr., with a current of air from an electric fan passing over them for a period of at least 2 hr., will cool the specimens to approximately room temperature. Specimens noticeably warm to the touch should not be used for the absorption test.

(*b*) *Saturation.*—The dry specimen shall be submerged, without preliminary partial immersion, in clean water (soft,

distilled, or rain water) at 15.5 to 30 C. (60 to 86 F.) for 24 hr. The specimen shall then be removed, the surface water wiped off with a damp cloth, and the specimen weighed. Weighing of any one specimen shall be completed within 5 min. after removing the specimen from the bath.

Calculations and Report

16. (*a*) The absorption of each specimen shall be calculated as follows:

$$\text{Absorption, per cent} = \frac{100(W_2 - W_1)}{W_1}$$

where:

W_1 = dry weight of the specimen, and
W_2 = saturated weight of the specimen after 24-hr. submersion in cold water.

(*b*) The average absorption of all the specimens tested shall be reported as the absorption of the lot of brick.

5-hr. Boiling Test

Test Specimens

17. The test specimens shall consist of half brick conforming to the requirements of Section 9. Five specimens shall be tested.

Procedure

18. (*a*) The specimen which has been saturated 24 hr. in the cold-water submersion bath shall be returned to the bath and subjected to a 5-hr. boiling test as described in Paragraph (*b*).

(*b*) The specimen shall be submerged in clear water (soft, distilled, or rain water) at 15.5 to 30 C. (60 to 86 F.) in such a manner that water can circulate freely on all sides of the specimen. The water shall be heated to boiling within 1 hr., boiled continuously for 5 hr., and then allowed to cool to 15.5 to 30 C. (60 to 86 F.) by natural loss of heat for not less than 16 nor more than 18 hr. The specimen shall then be removed, the surface water wiped off with a damp cloth, and the specimen weighed. Weighing of any one specimen shall be completed within 5 min. after removing the specimen from the bath.

NOTE.—If the tank is equipped with a drain so that water at 15.5 to 30 C. (60 to 86 F.) can be passed through the tank continuously and at such a rate that a complete change of water takes place in not more than 2 min., weighings can be made at the end of 1 hr.

Calculations and Report

19. (*a*) The absorption of each specimen shall be calculated as follows:

$$\text{Absorption, per cent} = \frac{100(W_3 - W_1)}{W_1}$$

where:

W_1 = dry weight of the specimen, and
W_3 = saturated weight of the specimen after 5-hr. submersion in boiling water.

(*b*) The average absorption of all the specimens tested shall be reported as the absorption of the lot of brick.

Saturation Coefficient

20. The saturation coefficient of each specimen shall be calculated as follows:

$$\text{Saturation coefficient} = \frac{W_2 - W_1}{W_3 - W_1}$$

where:

W_1 = dry weight of the specimen,
W_2 = saturated weight of the specimen after 24-hr. submersion in cold water, and
W_3 = saturated weight of the specimen after 5-hr. submersion in boiling water.

FREEZING AND THAWING

Apparatus

21. (*a*) *Compressor, Freezing Chamber, and Circulator.*—The design and capacity of the freezing apparatus shall be

such that the air of the freezing chamber shall not exceed 16 F. (− 9 C.) 1 hr. after introducing the maximum charge of brick, initially at a temperature not exceeding 90 F. (32 C.).

(*b*) *Trays and Containers.*—The brick shall be set on edge in shallow metal trays having an inside depth of $1\frac{1}{2} \pm \frac{1}{2}$ in., and of suitable strength and size so that the tray with its charge of frozen brick can be removed from the freezing chamber by one man.

(*c*) *Balance.*—The scale or balance used shall have a capacity of not less than 2000 g. and shall be sensitive to 0.5 g.

(*d*) *Drying Oven.*—The drying oven shall provide a free circulation of air through the oven and shall be capable of maintaining a temperature between 110 and 115 C. (230 to 239 F.).

(*e*) *Thawing Tank.*—A thawing tank shall be provided of such dimensions as to permit the complete submergence of the specimens in their trays. Adequate means shall be provided so that the water in the tank may be kept at a temperature of 75 ± 10 F. (24 ± 5.5 C.).

(*f*) *Drying Room.*—A room maintained at a temperature of 75 ± 15 F. (24 ± 8 C.), with a relative humidity between 30 and 70 per cent, and free from drafts shall be provided for the air-drying prescribed in alternate method B.

Test Specimens

22. The test specimens shall consist of half brick with approximately plane and parallel ends resulting from the flexure test. The specimens shall be free from shattering or unsoundness resulting from the flexure test or from the absorption tests (24-hr. submersion test and 5-hr. boiling test). Five specimens shall be tested.

Method A

Procedure

23. (*a*) The specimens that have previously been subjected to the 24-hr. submersion test and to the 5-hr. boiling test shall be dried as prescribed in Section 15 (*a*).

(*b*) When cool, the specimens shall be weighed on a scale or balance to the nearest 0.5 g.

(*c*) The test specimens shall be immediately submerged, without preliminary partial immersion, in the water of the thawing tank for 48 hr. before the start of the freezing-and-thawing test.

(*d*) The test specimens shall be stood on edge in the trays so that a space of at least $\frac{1}{2}$ in. separates the specimens. Sufficient water shall be poured into the trays so that each specimen stands in $\frac{1}{2}$-in. depth of water, and the trays and their contents shall be exposed to the temperature of the freezing chamber for 20 hr.

(*e*) The trays shall be removed from the freezing chamber after 20 hr. and shall be totally immersed with their contents in the water of the thawing tank for a time of 4 hr.

(*f*) The specimens shall be frozen by the procedure prescribed in Paragraph (*d*) and thawed by the procedure prescribed in Paragraph (*e*), three times. The test specimens shall then be dried by exposure for 24 hr. in the drying room followed by 72 hr. in the drying oven as prescribed in Paragraph (*a*). The test specimens shall be cooled, reweighed, immersed in water for 48 hr., and again subjected to three cycles of freezing and thawing in accordance with Paragraphs (*d*) and (*e*).

(*g*) Alternations of drying, weighing, and submersion in water for 48 hr. followed by 3 cycles of freezing and thawing shall be continued until a total of 51 cycles of freezing and thawing has been completed, unless the test specimen has lost more than 3 per cent of its weight, calculated on the basis of its original weight when dry, or has broken,

or has disintegrated, as judged by visual inspection, equivalent to more than 3 per cent loss before completion of 51 cycles.

Calculations and Report

24. The loss in weight after 51 cycles shall be calculated as a percentage of the original weight of the dried specimen; or the number of cycles required to cause breakage or 1 per cent and 3 per cent loss in weight shall be reported.

Method B

Procedure

25. (*a*) The initial drying of the test specimens shall be done as prescribed in Section 15 (*a*).

(*b*) The weight of the dried specimens shall be obtained as prescribed in Section 23 (*b*).

(*c*) The test specimens shall be immediately submerged, without preliminary partial immersion, in the water of the thawing tank for 4 hr. before the start of the freezing-and-thawing test.

(*d*) The test specimens shall be frozen by the procedure prescribed in Section 23 (*d*) and thawed by the procedure prescribed in Section 23 (*e*), five times. Following the 4-hr. thawing after the fifth freezing, the specimens shall be stored for 40 hr. on the floor of the drying room. They shall not be stacked or piled, and there shall be a space of at least 1 in. between any two specimens. Following this period of air-drying, the specimens shall be inspected, submerged in the water of the thawing tank for 4 hr., and again subjected to 5 cycles of freezing and thawing in accordance with Section 23 (*d*) and (*e*).

(*e*) Alternations of drying and submersion in water for 4 hr., followed by 5 cycles of freezing and thawing shall be continued until a total of 50 cycles of freezing and thawing has been completed, unless the test specimen has broken or appears to have lost more than 3 per cent of its original weight as judged by visual inspection.

(*f*) After completion of 50 cycles, or when the specimen is withdrawn from test as a result of evident disintegration, the specimen shall be dried in an oven as prescribed in Section 15 (*a*).

Calculations and Report

26. The loss in weight shall be calculated as a percentage of the original weight of the dried specimen, or the number of cycles causing breakage or withdrawal of the specimen shall be reported.

INITIAL RATE OF ABSORPTION (SUCTION)

Apparatus

27. The apparatus shall consist of the following:

(*a*) *Trays or Containers.*—Watertight trays or containers having an inside depth of not less than $\frac{1}{2}$ in. and of such length and width that an area of not less than 300 sq. in. of water surface is provided. The bottom of the tray shall provide a plane, horizontal upper surface, when suitably supported, so that an area not less than 8 in. in length by 6 in. in width will be level when tested by a spirit level.

(*b*) *Supports for Brick.*—Two noncorrodible metal supports consisting of bars between 5 and 6 in. in length, having triangular, half-round, or rectangular cross-sections such that the thickness (height) will be approximately $\frac{1}{4}$ in. The thickness of the two bars shall agree within 0.001 in. and, if the bars are rectangular in cross-section, their width shall not exceed $\frac{5}{16}$ in.

(*c*) *Means for Maintaining Constant Water Level.*—Suitable means for controlling the water level above the upper surface of the supports for the brick within plus or minus 0.01 in. (Note 1), including means for adding water to

the tray at a rate corresponding to the rate of removal by the brick undergoing test (Note 2). For use in checking the adequacy of the method of controlling the rate of flow of the added water, a reference brick or half brick shall be provided whose displacement in $\frac{1}{8}$ in. of water corresponds to the brick or half brick to be tested within plus or minus 2.5 per cent. The reference brick shall be completely submerged in water for not less than 3 hr. preceding its use as described in Section 3 (*d*).

NOTE 1.—A suitable means for obtaining this accuracy in control of the water level is provided by attaching to the end of one of the bars, two stiff metal wires which project upward and return, terminating in points one of which is $\frac{1}{8} - 0.01$ in. and the other $\frac{1}{8} + 0.01$ in. above the upper surface or edge of the bar. Such precise adjustment is obtainable by use of depth plates or a micrometer microscope. When the water level with respect to the upper surface or edge of the bar is adjusted so that the lower point "dimples" the water surface when viewed by reflected light and the upper point is not in contact with the water, the water level is within the limits specified. Any other suitable means for fixing and maintaining a constant depth of immersion may be used if equivalent accuracy is obtained. As an example of such other suitable means there may be mentioned the use of rigid supports movable with respect to the water level.

NOTE 2.—A rubber tube leading from a siphon or gravity feed and closed by a spring clip will provide a suitable manual control. The so-called "chicken feed" devices as a rule lack sensitivity and do not operate with the very small changes in water level permissible in this test.

(*d*) *Balance.*—A scale or balance having a capacity of not less than 3000 g. and sensitive to 0.5 g.

(*e*) *Drying Oven.*—A drying oven conforming to the requirements of Section 21 (*d*).

(*f*) *Constant Temperature Room.*—A room maintained at a temperature of 70 ± 2.5 F. (21 ± 1.4 C).

(*g*) *Timing Device.*—A suitable timing device, preferably a stop watch or stop clock, which shall indicate a time of 1 min. to the nearest second.

Test Specimens

28. The test specimens shall consist of whole brick or of half brick conforming to the requirements of Section 9. Five specimens shall be tested.

Procedure

29. (*a*) *Drying.*—The test specimens shall be dried in accordance with Section 15 (*a*).

(*b*) *Cooling.*—After drying, the test specimens shall be cooled in the constant temperature room (Section 27 (*f*)) by storage, unstacked, with separate placement for a period of at least 4 hr.

(*c*) *Measurement and Weighing.*—The length and width of the flatwise surface of the test specimen which will be in contact with the water shall be measured to the nearest 0.05 in. The specimen shall be weighed to the nearest 0.5 g.

(*d*) *Adjustment of Water Level.*—The tray for the absorption test shall be set up in the constant temperature room. The position of the tray shall be adjusted so that the upper surface of its bottom shall be level when tested by a spirit level and the saturated reference brick (Section 27 (*c*)) shall be set in place on top of the supports. Water shall be added until the water level is $\frac{1}{8} \pm 0.01$ in. above the top of the supports.

(*e*) *Absorption and Reweighing.* — After removal of the reference brick, the test brick shall be set in place flatwise, counting zero time as the moment of contact of the brick with the water. During the period of contact (1 min. ± 1 sec.) the water level shall be kept within the prescribed limits by adding water as required. At the end of 1 min. ± 1 sec., the brick shall be lifted from contact with the water, the surface water wiped off with a damp cloth, and the brick reweighed to the nearest 0.5

g. Wiping shall be completed within 10 sec. of removal from contact with water and weighing shall be completed within 2 min.

NOTE.—Placing of the brick in contact with the water shall be done quickly but without splashing. Setting the brick in position with a rocking motion will avoid the entrapping of air on its under surface. Brick with frogs or depressions in one flatwise surface shall be tested with the frog or depression uppermost.

Calculations and Report

30. (*a*) The difference in weight in grams between the initial and final weighings is the weight in grams of water absorbed by the brick during 1-min. contact with water. If the test specimen is a whole brick and the area of its flatwise surface (length times width) does not differ more than plus or minus 0.75 sq. in. (plus or minus 2.5 per cent) from 30 sq. in., the gain in weight in grams shall be reported as the initial rate of absorption in 1 min.

(*b*) If the test specimen is not a whole brick or the area of its flatwise surface differs more than plus or minus 0.75 sq. in. (plus or minus 2.5 per cent) from 30 sq. in., the gain in weight shall be calculated to the equivalent for 30 sq. in. as follows:

$$X = \frac{30\ W}{LB}$$

where:

X = gain in weight corrected to basis of 30 sq. in. flatwise area,
W = actual gain in weight of specimen in grams,
L = length of specimen in inches, and
B = width of specimen in inches.

The corrected gain in weight, X, shall be reported as the initial rate of absorption in 1 min.

(*c*) If the test specimen is a cored brick, the net area shall be calculated and substituted for LB in the formula given in Paragraph (*b*). The corrected gain in weight shall be reported as the initial rate of absorption in 1 min.

EFFLORESCENCE

Apparatus

31. The apparatus shall consist of the following:

(*a*) *Trays and Containers.* — Watertight shallow pans or trays made of metal or other material that will not provide soluble salts when in contact with distilled water containing leachings from brick. The pan shall be of such dimensions that it will provide not less than a 1-in. depth of water. Unless the pan provides an area such that the total volume of water is large in comparison with the amount evaporated each day, suitable apparatus shall be provided for keeping a constant level of water in the pan.

(*b*) *Drying Room.*—A drying room conforming to the requirements of Section 21 (*f*).

(*c*) *Drying Oven.*—A drying oven conforming to the requirements of Section 21 (*d*).

Test Specimens

32. (*a*) Ten dry full-size brick shall be tested.

(*b*) The ten specimens shall be sorted into five pairs, so that both specimens of each pair will have the same appearance, as nearly as possible.

Preparation of Specimens

33. The specimens shall be tested as received, except that any adhering dirt that might be mistaken for efflorescence shall be removed by brushing.

Procedure

34. (*a*) One specimen from each of the five pairs shall be set on end, partially immersed in distilled water to a depth of approximately 1 in. for 7 days in the drying room. When several specimens

are tested in the same container, the individual specimens shall be separated by a space of at least 2 in.

NOTE 1.—Testing specimens from different sources simultaneously in the same container is not recommended, because specimens with a considerable content of soluble salts may contaminate the salt-free specimens.

NOTE 2.—The pans or trays should be emptied and cleaned after each test.

(*b*) The second specimen from each of the five pairs shall be stored in the drying room without contact with water.

(*c*) At the end of 7 days, the first set of specimens shall be inspected and both sets shall then be dried in the drying oven for 3 days.

NOTE 3.—A drying period of 24 hr. is sufficient for the purpose of an efflorescence test. A 3-day period of drying prepares the specimens for the other tests (modulus of rupture, compressive strength, absorption, and initial rate of absorption) that may be specified.

Examination and Rating

35. After drying, each pair of specimens shall be examined and compared, observing the top and all four faces of each specimen. If there is no observable difference due to efflorescence, the rating shall be reported as "no efflorescence." If any difference due to efflorescence is noted, the specimens shall be viewed from a distance of 10 ft., under an illumination of not less than 50 foot-candles, by an observer with normal vision. If under these conditions no difference is noted, the rating shall be reported as "slightly effloresced." If a perceptible difference due to efflorescence is noted under these conditions, the rating shall be "effloresced." The appearance and distribution of the efflorescence shall be recorded.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: A74.1-1942

Standard Specifications for

STRUCTURAL CLAY LOAD-BEARING WALL TILE[1]

A.S.T.M. Designation: C 34 - 41

ADOPTED, 1936; REVISED, 1939, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 34; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. (*a*) These specifications cover structural clay load-bearing wall tile made from surface clay, shale, fire clay, or mixtures thereof. Two grades of tile are covered, as follows:

Grade LB X.—Suitable for general use in masonry construction and adapted for use in masonry exposed to weathering, provided they are burned to the normal maturity of the clay. They may also be considered suitable for the direct application of stucco.

Grade LB.—Suitable for general use in masonry where not exposed to frost action, or for use in exposed masonry where protected with a facing of 3 in. or more of stone, brick, terra cotta, or other masonry.

(*b*) If tile having a particular color, texture, or finish are desired, these features should be specified separately by the purchaser (see Explanatory Note 1).

Physical Properties

2. (*a*) Structural clay load-bearing wall tile shall conform to the requirements as to physical properties for the grade specified as prescribed in Table I.

TABLE I.—PHYSICAL REQUIREMENTS.

Grade	Absorption,[a] per cent			Compressive Strength (Based on Gross Area),[b] psi.			
				End-Construction Tile		Side-Construction Tile	
	Average of Five Tests	Individual Maximum	Individual Minimum	Minimum Average of Five Tests	Individual Minimum	Minimum Average of Five Tests	Individual Minimum
LB X	5 to 16	19	4	1400	1000	700	500
LB	5 to 25	28	4	1000	700	700	500

[a] The range in percentage absorption for tile delivered to any one job shall be not more than 12.

[b] Gross area of a unit shall be the total area including cells of a section perpendicular to the direction of loading. Re-entrant spaces are included in the gross area, unless these spaces are to be occupied in masonry by portions of adjacent units.

(*b*) Tile of grade LB X shall be accepted under all conditions in lieu of grade LB.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-15 on Manufactured Masonry Units.

[2] Prior to their present adoption as standard, these specifications were published as tentative from 1921 to 1926, being revised in 1924 and 1926. They were adopted in 1926, revised in 1927, 1930, and 1931, but withdrawn and replaced in 1934 by C 34 - 34 T which were published as tentative from 1933 to 1936, being revised in 1934, 1935, and 1936.

(*c*) End-construction tile are tile designed to be placed in the wall with axes of the cells vertical. Side-construction tile are tile designed to be placed in the wall with the axes of the cells horizontal. Where end-construction tile are used on the side they shall conform to the requirements of side-construction tile and *vice versa*.

(*d*) Bonding tile shall be so designed as to provide recesses for header brick courses when laid up in brick-faced walls.

Number of Cells

3. (*a*) Load-bearing tile shall conform to the following requirements as to minimum number of cells[a] in the direction of wall thickness (see Explanatory Note 2 for approximate weights of tile):

Nominal Horizontal Thickness of Tile as Laid in Wall, in.	Minimum Number of Cells[a] in Direction of Wall Thickness
4	1
6	2
8	2
10	2
12	3

[a] Cells are hollow spaces enclosed within the perimeter of the exterior shells, and having a minimum dimension of not less than $\frac{1}{2}$ in. and a cross-sectional area of not less than 1 sq. in.

(*b*) In double-shell tile, the two voids between exterior and interior shells on either side of the tile shall be considered as one cell in thickness of wall when their combined width is not less than $\frac{1}{2}$ in., provided the short webs between the inner and outer shells are not greater in number or thickness than the long transverse webs holding the inner shells.

(*c*) Re-entrant spaces not less than 1 in. in depth and not less than 1 sq. in. in area, which form cells when the units are laid in the walls, shall be considered as cells in the direction of wall thickness, but not in the units.

Shell and Web Thickness

4. (*a*) The average over-all thickness of the shells, measured between the inner and extreme outer surfaces of end-construction load-bearing tile, shall be not less than $\frac{3}{4}$ in., except that in double-shell tile the combined average over-all thickness of the inner and outer shell shall be not less than $\frac{3}{4}$ in. The thickness of the webs shall be not less than $\frac{1}{2}$ in.

(*b*) The average over-all thickness of the shells, measured between the inner and extreme outer surfaces of side-construction load-bearing tile, shall be not less than $\frac{3}{8}$ in., except that in double-shell tile the combined average over-all thickness of the inner and outer shell shall be not less than $\frac{3}{4}$ in. The thickness of the webs shall be not less than $\frac{1}{2}$ in.

(*c*) The width of any cell in side-construction tile, measured in the direction of wall thickness, shall not exceed four and one half times the average over-all thickness of either the upper or lower bearing shells. If no cell in side-construction tile, measured in the direction of the wall thickness, exceeds four times the average over-all thickness of either the upper or lower bearing shells, the requirements for minimum shell and web thickness specified in Paragraph (*b*) shall be waived.

(*d*) The thickness of bonding and other types of tile manufactured for use in combination with brick or other materials may vary from the nominal thicknesses indicated in Section 3, as required by construction requirements.

Permissible Variations in Dimensions

5. No over-all dimension shall vary more than 3 per cent over or under the specified dimension for any form or size of tile.

Scoring

6. Surfaces of all tile intended for the direct application of plaster or stucco shall be scratched or scored. When

scored, each groove shall be not less than $\frac{1}{8}$ nor more than $\frac{1}{4}$ in. in depth, nor more than 1 in. in width. The area covered by the grooves shall not exceed 50 per cent of the area of the scored faces.

Marking

7. All load-bearing wall tile shall bear the word "Load-Bearing," or LB X or LB, and the name, initials, or trade mark of the manufacturer. These marks shall be indented on the exterior of the tile and shall be legible.

Visual Inspection

8. (*a*) All tile shall be reasonably free from laminations and from cracks, blisters, surface roughness, and other defects that would interfere with the proper setting of the tile or impair the strength or permanence of the construction.

(*b*) When unscored (smooth) tile are specified, the requirements, except as to scoring, shall be the same as for scored tile. If closer tolerances on dimensions and on freedom from belt marks, chips, surface discolorations, and roughness are desired, such requirements shall be clearly defined in supplementary specifications.

Rejection

9. In case the shipment fails to conform to the requirements for the grade specified, the manufacturer may sort it, and new specimens shall be selected by the purchaser from the retained lot and tested at the expense of the manufacturer. In case the second set of specimens fails to meet the requirements, the entire lot shall be rejected.

Expense of Tests

10. Except as specified in Section 9, and unless otherwise agreed, the expense of inspection and testing shall be borne by the purchaser.

Sampling and Testing

11. (*a*) The purchaser or his authorized representative shall be accorded proper facilities for sampling and inspection of units both at the place of manufacture and at the site of the work. At least ten days from the time of sampling should be allowed for completion of the tests.

(*b*) Tile shall be sampled and tested in accordance with the Standard Methods of Sampling and Testing Structural Clay Tile (A.S.T.M. Designation: C 112) of the American Society for Testing Materials.[3]

EXPLANATORY NOTES

NOTE 1.—Color of tile varies with the type of clay used and degree of burning; hence, it cannot be taken as indicative of classification until after it has been related to absorption and strength by actual tests.

NOTE 2.—The following average weights of structural clay load-bearing wall tile are given only as information:

Nominal Horizontal Thickness of Tile as Laid in Wall, in.	Average Weight, lb. per sq. ft. of tile[a]
4	20
6	30
8	36
10	42
12	52

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

[a] The weights given in the above table are for scored tile. If any of the faces are not scored, the weights are increased 1 lb. per sq. ft. of unscored area.

APPROVED AS
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Standard Specifications for

STRUCTURAL CLAY NON-LOAD-BEARING TILE[1]

A.S.T.M. Designation: C 56 - 41

ADOPTED, 1936; REVISED, 1939, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 56; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. (*a*) These specifications cover structural clay non-load-bearing tile (partition, fireproofing, and furring) made from surface clay, shale, fire clay, or mixtures thereof. One grade of tile is covered, designated grade NB.

(*b*) Fireproofing tile intended for use in load-bearing masonry shall conform to the Standard Specifications for Structural Clay Load-Bearing Wall Tile (A.S.T.M. Designation: C 34) of the American Society for Testing Materials.[3]

(*c*) If tile having a particular color (Note), texture, or finish are desired, these features should be specified separately by the purchaser.

NOTE.—Color of tile varies with the type of clay used and degree of burning; hence, it cannot be taken as indicative of classification until after it has been related to absorption by actual tests.

Absorption

2. Structural clay non-load-bearing tile shall conform to the following requirements as to absorption:

Grade	Absorption,[a] per cent		
	Average of Five Tests	Individual Maximum	Individual Minimum
NB.....	5 to 25	28	4

[a] The range in percentage absorption for tile delivered to any one job shall be not more than 12.

Number of Cells and Weights

3. (*a*) Partition and split furring tile shall conform to the requirements prescribed in Table I as to total number of cells, number of cells in the direction of wall thickness, and dry weights per square foot of tile.

(*b*) The requirements for minimum weights of structural clay non-load-bearing tile shall be waived if the overall thickness of the shells, measured between the inner and extreme outer sur-

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-15 on Manufactured Masonry Units.

[2] Prior to their present adoption as standard, these specifications were published as tentative from 1924 to 1930, being revised in 1926, 1927, 1928, and 1930. They were adopted in 1930, revised in 1931, but withdrawn and replaced in 1934 by C 56 - 34 T which was published as tentative from 1933 to 1936, being revised in 1934, 1935, and 1936.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

faces, is not less than $\frac{5}{8}$ in. and the thickness of the webs is not less than $\frac{1}{2}$ in.

TABLE I.—NUMBER OF CELLS AND WEIGHT OF TILE.

Dimensions, in.	Minimum Number of Cells		Minimum Weight,[a] lb. per sq. ft. of tile	
	In Unit	In Direction of Wall Thickness	Average	Individual
PARTITION TILE				
2 by 12 by 12	3	1	14	13
3 by 12 by 12	3	1	15	14
4 by 12 by 12	3	1	16	15
6 by 12 by 12	3	1	22	21
6 by 12 by 12	4	2	25	24
8 by 12 by 12	4	2	30	28
10 by 12 by 12	4	2	35	33
12 by 12 by 12	4	2	40	38
SPLIT FURRING TILE				
1½ by 12 by 12	3	..	7½	7
2 by 12 by 12	3	..	8	7½

[a] The weights given in the table are for scored tile. If any of the faces are unscored, the weights shall be increased 0.5 lb. per square foot of unscored area.

Permissible Variations in Dimensions

4. No over-all dimension shall vary more than 3 per cent over or under the specified dimension for any form or size of tile.

Fireproofing Tile

5. Tile intended for use in fireproofing structural members shall be of such sizes and shapes that they can be erected to cover completely the exposed surfaces of the members.

Scoring

6. Surfaces of all tile intended for the direct application of plaster or stucco shall be scratched or scored. When scored, each groove shall be not less than $\frac{1}{8}$ nor more than $\frac{3}{16}$ in. in depth, nor more than 1 in. in width. The area covered by the grooves shall not exceed 50 per cent of the area of the scored faces.

Marking

7. All non-load-bearing tile shall bear the name, initials, or trade mark of the manufacturer. These marks shall be indented on the exterior of the tile and shall be legible.

Visual Inspection

8. (*a*) All tile shall be reasonably free from laminations and from cracks, blisters, surface roughness, and other defects that would interfere with the proper setting of the tile or impair the strength or permanence of the construction.

(*b*) When unscored (smooth) tile are specified, the requirements, except as to scoring, shall be the same as for scored tile. If closer tolerances on dimensions and on freedom from belt marks, chips, surface discolorations, and roughness are desired, such requirements shall be clearly defined in supplementary specifications.

Rejection

9. In case the shipment fails to conform to the requirements, the manufacturer may sort it, and new specimens shall be selected by the purchaser from the retained lot and tested at the expense of the manufacturer. In case the second set of specimens fails to meet the requirements, the entire lot shall be rejected.

Expense of Tests

10. Except as specified in Section 9, and unless otherwise agreed, the expense of inspection and testing shall be borne by the purchaser.

Sampling and Testing

11. (*a*) The purchaser or his authorized representative shall be accorded proper facilities for sampling and inspection of units both at the place of manu-

facture and at the site of the work. At least ten days from the time of sampling should be allowed for completion of the tests.

(*b*) Tile shall be sampled and tested in accordance with the Standard Methods of Sampling and Testing Structural Clay Tile (A.S.T.M. Designation: C 112) of the American Society for Testing Materials.[3]

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AMERICAN STANDARD
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ASA NO.: A77.1-1942

Standard Specifications for

STRUCTURAL CLAY FLOOR TILE[1]

A.S.T.M. Designation: C 57 – 39

ADOPTED, 1936; REVISED, 1939.[2]
Reapproved in 1944 Without Change.

This Standard of the American Society for Testing Materials is issued under the fixed designation C 57; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. (*a*) These specifications cover structural clay floor tile made from surface clay, shale, fire clay, or mixtures thereof. Two grades of tile are covered, designated grade *FT1* and grade *FT2*. Both grades are suitable for use in flat or segmental arches or in combination tile and concrete ribbed-slab construction.

(*b*) If tile having a particular color (Note), texture, or finish are desired, these features should be specified separately by the purchaser.

NOTE—Color of tile varies with the type of clay used and degree of burning; hence, it cannot be taken as indicative of classification until after it has been related to absorption and strength by actual tests.

Physical Properties

2. (*a*) Structural clay floor tile shall conform to the requirements as to physical properties for the grade specified as prescribed in Table I.

(*b*) End-construction floor tile are tile designed to resist forces applied in a direction parallel to axes of the cells. Side-construction floor tile are tile designed to resist forces applied in a direction perpendicular to axes of the cells. Where end-construction tile are used on the side, they shall conform to the requirements of side-construction tile and *vice versa*.

TABLE I.—PHYSICAL REQUIREMENTS.

Grade	Absorption,[a] per cent			Compressive Strength (Based on Net Area),[b] psi.			
				End-Construction Tile		Side-Construction Tile	
	Average of Five Tests	Individual Maximum	Individual Minimum	Minimum Average of Five Tests	Individual Minimum	Minimum Average of Five Tests	Individual Minimum
FT1	5 to 25	25	4	3200	2250	1600	1100
FT2	5 to 25	25	4	2000	1400	1200	850

[a] The range in percentage absorption for tile delivered to any one job shall be not more than 12.
[b] Net area of a unit shall be taken as the area of solid material in shells and webs actually carrying stresses in a direction parallel to the direction of loading.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-15 on Manufactured Masonry Units.

[2] Prior to their present adoption as standard, these specifications were published as tentative from 1924 to 1927, being revised in 1927. They were adopted in 1927, revised in 1930 and 1931, but withdrawn and replaced in 1934 by C 57 – 34 T which was published as tentative from 1933 to 1936, being revised in 1934, 1935, and 1936.

Number of Cells and Weights

3. (*a*) Flat and segmental arch tile shall conform to the requirements prescribed in Table II as to number of cells in the depth of arch, and average dry weights per square foot of tile.

TABLE II.—NUMBER OF CELLS AND WEIGHT OF TILE FOR USE IN ARCHES.

Depth of Arch, in.	Minimum Number of Cells in Depth of Arch	Average Dry Weight, lb. per sq. ft. of tile	
		Minimum	Maximum
FLAT ARCH TILE			
6	1	25	29
7	1	28	34
8	2	30	36
9	2	31	37
10	2	33	39
11	2	36	43
12	2	38	45
13	3	41	48
14	3	44	52
15	3	45	53
16	3	49	58
SEGMENTAL ARCH TILE			
6	2	28	34
8	2	34	40

(*b*) Tile designed to resist stresses in combination tile and concrete ribbed-slab construction shall conform to the requirements prescribed in Table III as to number of cells in the direction of depth of slab, and average dry weights per square foot of tile.

(*c*) The requirements for minimum weights of structural clay floor tile shall be waived if the over-all thickness of the shells, measured between the inner and extreme outer surfaces, is not less than $\frac{5}{8}$ in. and the thickness of the webs is not less than $\frac{1}{2}$ in.

Permissible Variations in Dimension

4. No over-all dimension shall vary more than 3 per cent over or under the specified dimension for any form or size of tile.

Visual Inspection

5. All tile shall be reasonably free from laminations and from cracks, blisters, surface roughness, and other defects that would interfere with the proper setting of the tile or impair the strength or permanence of the construction.

TABLE III.—NUMBER OF CELLS AND WEIGHT OF TILE FOR SLAB CONSTRUCTION.

Thickness of Units, in.	Minimum Number of Cells[a] in Thickness of Tile	Average Dry[b] Weight, lb. per sq. ft. of tile	
		Minimum	Maximum
3	1	13.5	16.5
4	1	14.5	17.5
5	1	17.0	21.0
5	2	20.5	25.5
6	1	20.0	24.0
6	2	22.5	27.5
7	1	21.5	26.5
7	2	24.5	29.5
8	1	24.5	29.5
8	2	26.0	32.0
9	2	28.0	34.0
9	3	32.5	39.5
10	2	29.5	36.5
10	3	34.0	42.0
12	2	36.0	44.0
12	3	40.5	49.5

[a] Cells are hollow spaces enclosed within the perimeter of the exterior shells, and having a minimum dimension of not less than $\frac{1}{4}$ in. and a cross-sectional area of not less than 1 sq. in.

[b] The weights given in the table are for scored tile. If any of the faces are unscored, the weights shall be increased 0.5 lb. per sq. ft. of unscored area.

Scoring

6. Surfaces of all tile intended for the direct application of plaster shall be scratched or scored. When scored, each groove shall be not less than $\frac{1}{4}$ nor more than $\frac{3}{16}$ in. in depth, and not more than 1 in. in width. The area covered by the grooves shall not exceed 50 per cent of the area of the scored faces.

Marking

7. All floor tile shall bear the name, initials, or trade mark of the manufacturer. These marks shall be indented on the exterior of the tile and shall be legible.

Rejection

8. In case the shipment fails to conform to the requirements for the grade specified, the manufacturer may sort it, and new specimens shall be selected by the purchaser from the retained lot and tested at the expense of the manufacturer. In case the second set of specimens fails to meet the requirements, the entire lot shall be rejected.

Expense of Tests

9. Except as specified in Section 8, and unless otherwise agreed, the expense of inspection and testing shall be borne by the purchaser.

Sampling and Testing

10. (*a*) The purchaser or his authorized representative shall be accorded proper facilities for sampling and inspection of units both at the place of manufacture and at the site of the work. At least ten days from the time of sampling should be allowed for completion of the tests.

(*b*) Tile shall be sampled and tested in accordance with the Standard Methods of Sampling and Testing Structural Clay Tile (A.S.T.M. Designation: C 112) of the American Society for Testing Materials.[3]

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

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Standard Methods of

SAMPLING AND TESTING STRUCTURAL CLAY TILE[1]

A.S.T.M. Designation: C 112 - 36

ADOPTED, 1936.[2]

Reapproved in 1944 Without Change.

This Standard of the American Society for Testing Materials is issued under the fixed designation C 112; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods cover procedures for the sampling and testing of structural clay tile for weight determinations, compressive strength, absorption, and freezing and thawing.

Definitions of Terms

2. The Standard Definitions of Terms Relating to Methods of Testing (A.S.T.M. Designation: E 6) of the American Society for Testing Materials[3] shall be considered as applying to the terms used in these methods.

SAMPLING

Selection of Test Specimens

3. For purpose of tests, full-size tile shall be selected by the purchaser or by his authorized representative. Specimens shall be representative of the whole lot of tile from which they are selected.

Number of Specimens

4. For the weight determination, compressive strength, absorption, and freezing-and-thawing tests, at least five tile shall be selected from each kiln or from each lot of 100 tons or fraction thereof. In no case shall less than five tile be taken. Additional specimens may be taken at the discretion of the purchaser.

Identification

5. Each specimen shall be marked so that it may be identified at any time. Markings shall not cover more than 5 per cent of the superficial area of the specimen.

WEIGHT DETERMINATION

Test Specimens

6. Five full-size tile shall be tested.

Procedure

7. The five tile specimens, if not in kiln-dry condition, shall be dried to constant weight in a ventilated oven at 110 to 115 C. (230 to 239 F.) and shall then be weighed separately. The balance used shall be accurate to within 0.5 per cent of the weight of the smallest specimen tested.

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee C-15 on Manufactured Masonry Units.

[2] Prior to adoption as standard, these methods were published as tentative from 1934 to 1936, being revised in 1935 and 1936.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Calculations and Reports

8. (*a*) The weight per unit area of a tile specimen shall be calculated by dividing the total weight in pounds by the average area in square feet of the two faces of the tile as laid in the masonry.

(*b*) The results shall be reported separately for each tile with the average for the five tile.

COMPRESSIVE STRENGTH

Test Specimens

9. Five dry full-size tile shall be tested.

Capping Test Specimens

10. (*a*) Bearing surfaces of the test specimens and portions adjoining them which are liable to absorb water from the plastic capping mortar shall be coated with shellac and allowed to dry. A quantity of plastic mortar, made of unretarded gypsum (plaster of Paris) mixed with sufficient water to spread evenly, shall be placed on a plane surface which has been coated with oil. The surface to be capped shall be placed on this mortar, and the specimen, so held that its axis is at right angles to the capping surface, shall be firmly pressed down with a single motion.

(*b*) The average thickness of the cap after the extruded capping mortar has been removed and the edges trued shall be not more than $\frac{1}{8}$ in. Patching of caps after setting shall not be permitted. Imperfect caps shall be removed and replaced with new ones.

(*c*) When specimens have portions of bearing surfaces in different planes, these surfaces shall be made plane and approximately parallel by means of a filling of mortar composed of one part by weight of a quick-hardening cement, conforming to the requirements for type III cement in the Standard Specifications for Portland Cement (A.S.T.M. Designation: C 150) of the American Society for Testing Materials,[2] and two parts of sand. The filling shall be aged for 1 day, after which the specimens shall be capped as specified in Paragraph (*a*).

(*d*) The caps shall be aged at least 2 days before the specimens are tested.

Procedure

11. (*a*) *Position of Specimens.*—All specimens shall be tested in a position such that the load is applied in the same direction as in service.

(*b*) *Testing Machine.*—The testing machine shall conform to the requirements of the Standard Methods of Verification of Testing Machines (A.S.T.M. Designation: E 4) of the American Society for Testing Materials.[3]

(*c*) *Bearing Block.*—The upper bearing shall be a spherically seated, hardened metal block firmly attached at the center of the upper head of the machine. The center of the sphere shall lie at the center of the surface of the block in contact with the specimen. The block shall be closely held in its spherical seat, but shall be free to turn in any direction. The diameter of the bearing surface shall be at least 6 in. A hardened metal bearing block shall be used beneath the specimen to minimize wear of the lower platen of the machine. The bearing block surfaces intended for contact with the specimen should have a hardness not less than C60 Rockwell number (Brinell number 620). These surfaces shall not depart from plane surfaces by more than 0.001 in. When the bearing area of the spherical bearing block is not sufficient to cover the area of the specimen, a steel plate with surfaces machined to true planes within plus or minus 0.001 in., and with a thickness equal to at least one-third of the distance

from the edge of the spherical bearing to the most distant corner, shall be placed between the spherical bearing block and the capped specimen.

(*d*) *Speed of Testing.*—The speed of the moving head of the testing machine shall be not more than 0.05 in. per min.

Calculations and Report

12. (*a*) The compressive strength of load-bearing wall tile shall be taken as the maximum load in pounds, divided by the gross cross-sectional area of the tile in square inches. The gross area of a tile is the total area of a section perpendicular to the direction of the load, including areas within cells and within re-entrant spaces unless these spaces are to be occupied in the masonry by portions of adjacent masonry.

(*b*) The compressive strengths of floor tile and non-load-bearing tile shall be taken as the maximum load in pounds, divided by the net cross-sectional area in square inches. The net area of a tile is the area of the fired clay in the section of minimum area taken perpendicular to the direction of the load.

(*c*) The results shall be reported separately for each tile, with the average for the five tile.

ABSORPTION

Test Specimens

13. The specimens for the absorption test shall consist of five tile or three representative pieces from each of these five tile. If small pieces are used, two shall be taken from the shell and one from an interior web, the weight of each piece to be not less than 227 g. (0.5 lb.). The specimens shall have had their rough edges or loose particles ground off and be free from cracks from the failure of the tile in compression, where taken from tile which have been subjected to strength tests.

Accuracy of Weighings

14. The balance used shall be sensitive to within 0.2 per cent of the weight of the smallest specimen tested.

Procedure

15. (*a*) *Drying.*—All specimens shall be dried to constant weight in a ventilated oven at 110 to 115 C. (230 to 239 F.)

(*b*) *Saturation.*—After obtaining the dry weight of the specimens they shall be immersed in soft, distilled, or rain water, the temperature raised to the boiling point, and the water boiled continuously for 1 hr. The specimens shall then be allowed to cool in the water to room temperature.

(*c*) *Weighing.*—After saturation, the specimens shall be removed from the water and allowed to drain for not more than 1 min. The superficial water shall then be removed from the specimens with a damp cloth, after which they shall be weighed immediately.

Calculations and Report

16. The absorption shall be calculated as a percentage of the initial dry weight, carried to the nearest first decimal place. The results shall be reported separately for each tile, with the average for the five tile.

FREEZING AND THAWING

Preparation of Specimens

17. The specimens for the freezing-and-thawing test shall consist of five tile or of a cell not less than 4 in. in length sawed from each of these five tile. The specimens shall be saturated by immersion in cold water for at least 72 hr. prior to starting the freezing.

Procedure

18. Any practical method of obtaining alternate freezings and thawings may

be used, the freezings always to be made with fully saturated specimens and the time and temperature to be such as to insure full freezing and thawing throughout the specimen. The initial weighing and all weighings for loss of weight shall be made on dry specimens.

Accuracy of Weighings

19. The balance used shall be sensitive to within 0.1 per cent of the weight of the smallest specimen tested.

Interpretation of Results

20. Failure under the freezing-and-thawing treatment shall be considered to be reached when:

(*a*) The specimens show superficial disintegration or spalling with loss of weight of more than 5 per cent of the initial weight,

(*b*) The specimens are badly cracked, or

(*c*) The specimens show evident loss of structural strength.

Standard Definitions of

TERMS RELATING TO STRUCTURAL CLAY TILE[1]

A.S.T.M. Designation: C 43 - 36

ADOPTED, 1936.[2]

Reapproved in 1944 Without Change.

This Standard of the American Society for Testing Materials is issued under the fixed designation C 43; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

TILE

Structural Clay Tile.—Hollow burned-clay masonry building units with parallel cells.

NOTE.—The term "tile" is understood, within the meaning of these definitions, to mean "structural clay tile." The term "terra cotta," which is applied to ornamental building units of burned clay, should not be used to designate structural clay tile.

Load-Bearing Tile.—Tile for use as load-bearing structural units in masonry walls.

Fireproofing Tile.—Tile for use as a protection for structural members against fire.

Partition Tile.—Tile for use in building interior partitions, subdividing areas into rooms, or similar construction, and carrying no superimposed loads.

Furring Tile.—Tile for lining the inside of walls and carrying no superimposed loads.

Foundation Tile.—Tile for use as load-bearing structural units in foundation walls.

Floor Tile.—Tile for use as structural units in floor and roof construction.

Bonding Tile.—Tile designed to provide recesses for header brick in brick-faced walls.

Book Tile.—Tile with tongue and groove edges resembling a book in shape.

GENERAL TERMS

Side-Construction Tile.—Tile designed to receive its principal stress at right angles to the axes of the cells.

End-Construction Tile.—Tile designed to receive its principal stress parallel to the axes of the cells.

Salt-Glazed Tile.—Tile with vitreous glaze on its surface produced by burning salt in the kiln at the temperature used in finishing the burning.

Porous Structural Clay Tile.—Tile in which the natural porosity of the clay has been increased by the admixture of other ingredients.

RAW MATERIALS

Shale.—A thinly stratified, consolidated, sedimentary clay[3] with well-marked cleavage parallel to the bedding.

Fire Clay.—A sedimentary clay[3] of low flux content.

NOTE.—Fire clay is usually associated with coal measures.

[1] Under the standardization procedure of the Society, these definitions are under the jurisdiction of the A.S.T.M. Committee C-15 on Manufactured Masonry Units.

[2] Prior to their present adoption as standard, these definitions were published as tentative from 1921 to 1924, being revised in 1923 and 1926. They were adopted in 1924, revised in 1931, but withdrawn and republished as tentative from 1933 to 1936, being revised in 1934.

[3] The definitions for the terms "shale," "fire clay" and "surface clay" are based upon the following definition of the term "clay."

Clay.—An earthy or stony mineral aggregate consisting essentially of hydrous silicates of alumina, plastic when sufficiently pulverized and wetted, rigid when dry, and vitreous when fired to a sufficiently high temperature.

Surface Clay.—An unconsolidated, unstratified clay,[3] occurring on the surface.

DESIGNATION OF DIMENSIONS

Thickness.—That dimension designed to lie at right angles to the face of the wall, floor, or other member in which it is used.

Width.—That dimension measured at right angles to the direction of its thickness and length.

Length.—That dimension measured between the cut ends.

NOTE 1.—In practice, the first dimension given represents thickness; the second, width; the third, length.

NOTE 2.—Gross area is taken as total area enclosed by the outside dimensions of the unit in a direction perpendicular to that in which load is carried.

NOTE 3.—Net area shall be taken as the area of solid material in shells and webs actually carrying stresses through the units in a direction parallel to the direction of loading.

PARTS, OPENINGS, AND SURFACE FEATURES

Shell.—The outer walls.

Webs.—The partitions dividing it into cells.

Cells.—Hollow spaces enclosed within the perimeter of the exterior shells and having a minimum dimension of not less than ½ in. and a cross-sectional area of not less than 1 sq. in.

Scoring.—The grooves formed in the exterior faces of the shells to increase the bond of mortar, plaster, or stucco.

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Standard Specifications for

HOLLOW LOAD-BEARING CONCRETE MASONRY UNITS[1]

A.S.T.M. Designation: C 90 – 44

ADOPTED, 1936; REVISED, 1939, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 90; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover hollow load-bearing concrete masonry wall units made from portland cement and suitable aggregates such as sand, gravel, crushed stone, bituminous or anthracite cinders, burned clay or shale, and blast-furnace slag.[3]

NOTE.—When a particular surface texture, finish, or uniformity of color is desired, these features should be specified separately by the purchaser.

Cinder Aggregate

2. The combustible content present in cinder aggregate shall not exceed 35 per cent of the weight of the aggregate.

Physical Requirements

3. At the time of delivery to the site of the work the units shall conform to the physical requirements prescribed in Table I.

TABLE I.—PHYSICAL REQUIREMENTS.

Minimum Face Shell Thickness, in.	Compressive Strength, min., psi. (Average Gross Area)		Water Absorption, max., lb. per cu. ft.	Moisture Content, max., per cent
	Average of 5 Units	Individual Unit	Average of 5 Units	Average of 5 Units
1¼ or over:				
Grade A[a]	1000	800	15	40
Grade B[b]	700	600	..	40
Under 1¼ and over ¾	1000	800	15	40

[a] For use in exterior walls below grade, and for unprotected exterior walls above grade.

[b] For general use above grade where protected from the weather with two coats of portland cement paint or other satisfactory waterproofing treatment approved by the purchaser.

Permissible Variations in Dimensions

4. No over-all dimension shall vary more than 3 per cent over or under the specified dimension for any form of unit.

Visual Inspection

5. (*a*) All units shall be sound and free from cracks or other defects that would interfere with the proper placing of the unit or impair the strength or permanence of the construction.

(*b*) Units that are intended to serve as a base for plaster or stucco shall have a sufficiently rough surface to afford good bond.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-15 on Manufactured Masonry Units.

[2] Prior to adoption as standard, these specifications were published as tentative from 1931 to 1936, being revised in 1933 and 1934.

[3] When fire tests are required they shall be conducted in accordance with the Standard Methods of Fire Tests of Building Construction and Materials (A.S.T.M. Designation: C 19), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Marking

6. All units shall bear a distinctive mark of the manufacturer or shall be otherwise readily identified as to origin.

Sampling and Testing

7. (*a*) The purchaser or his authorized representative shall be accorded proper facilities to inspect and sample the units at the place of manufacture from the lots ready for delivery. At least ten days should be allowed for completion of the tests.

(*b*) Units shall be sampled and tested in accordance with the Standard Methods of Sampling and Testing Concrete Masonry Units (A.S.T.M. Designation: C 140) of the American Society for Testing Materials.[4]

Rejection

8. In case the shipment fails to conform to the requirements, the manufacturer may sort it, and new specimens shall be selected by the purchaser from the retained lot and tested at the expense of the manufacturer. In case the second set of specimens fails to conform to the test requirements, the entire lot shall be rejected.

Expense of Tests

9. Except as specified in Section 8, and unless otherwise agreed, the expense of inspection and testing shall be borne by the purchaser.

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[4] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

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Standard Specifications for

HOLLOW NON-LOAD-BEARING CONCRETE MASONRY UNITS[1]

A.S.T.M. Designation: C 129 - 39

ADOPTED, 1939.[2]

Reapproved in 1944 Without Change.

This Standard of the American Society for Testing Materials is issued under the fixed designation C 129; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover hollow non-load-bearing concrete masonry wall units made from portland cement and suitable aggregates such as sand, gravel, crushed stone, bituminous or anthracite cinders, burned clay or shale, and blast-furnace slag.

NOTE.—When a particular surface texture, finish, or uniformity of color is desired, these features should be specified separately by the purchaser.

Cinder Aggregate

2. The combustible content present in cinder aggregate shall not exceed 35 per cent of the weight of the aggregate.

Physical Requirements

3. At the time of delivery to the site of the work the units shall conform to the physical requirements prescribed in Table I.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-15 on Manufactured Masonry Units.

[2] Prior to adoption as standard, these specifications were published as tentative from 1937 to 1939, being revised in 1938 and 1939.

Dimensions and Permissible Variations

4. (*a*) Minimum face shell thickness shall be not less than $\frac{1}{2}$ in.

(*b*) No over-all dimension shall vary more than 3 per cent over or under the specified dimension for any form of unit.

Visual Inspection

5. (*a*) All units shall be sound and free from cracks or other defects that would interfere with the proper placing of the unit.

TABLE I.—PHYSICAL REQUIREMENTS.

Compressive Strength, min., psi. (Average Gross Area)		Moisture Content, max., per cent of total absorption
Average of 5 Units	Individual Unit	Average of 5 Units
350	300	40

(*b*) Units that are intended to serve as a base for plaster or stucco shall have a sufficiently rough surface to afford good bond.

Marking

6. All units shall bear a distinctive mark of the manufacturer or shall be otherwise readily identified as to origin.

Sampling and Testing

7. (*a*) The purchaser or his authorized representative shall be accorded proper facilities to inspect and sample the units at the place of manufacture from the lots ready for delivery. At least ten days should be allowed for completion of the tests.

(*b*) Units shall be sampled and tested in accordance with the Standard Methods of Sampling and Testing Concrete Masonry Units (A.S.T.M. Designation: C 140) of the American Society for Testing Materials.[3]

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Rejection

8. In case the shipment fails to conform to the requirements, the manufacturer may sort it, and new specimens shall be selected by the purchaser from the retained lot and tested at the expense of the manufacturer. In case the second set of specimens fails to conform to the test requirements, the entire lot shall be rejected.

Expense of Tests

9. Except as specified in Section 8, and unless otherwise agreed, the expense of inspection and testing shall be borne by the purchaser.

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BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: A81.1-1942

Standard Specifications for

SOLID LOAD-BEARING CONCRETE MASONRY UNITS[1]

A.S.T.M. Designation: C 145 - 40

ADOPTED, 1940.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 145; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover solid load-bearing concrete masonry wall units (concrete block or tile units with 75 per cent or more net area) made from portland cement and suitable aggregates such as sand, gravel, crushed stone, bituminous or anthracite cinders, burned clay or shale, and blast-furnace slag.[3] Two grades of units are covered: grade A and grade B.

NOTE.—When a particular surface texture, finish, or uniformity of color is desired, these features should be specified separately by the purchaser.

Cinder Aggregate

2. The combustible content present in cinder aggregate shall not exceed 35 per cent of the weight of the aggregate.

Physical Requirements

3. (*a*) At the time of delivery to the site of the work the units shall conform to the physical requirements for the grades specified, as prescribed in Table I.

(*b*) Units that are not exposed to the weather or soil in the finished work need not conform to the absorption requirement prescribed in Table I.

TABLE I.—PHYSICAL REQUIREMENTS.

	Compressive Strength, min., psi. (Average Gross Area)		Water Absorption, max., lb. per cu. ft.	Moisture Content, max., per cent
	Average of 5 Units	Individual Unit	Average of 5 Units	Average of 5 Units
Grade A.....	1800	1600	15	40
Grade B.....	1200	1000	15	40

NOTE.—The above classification is based on strength and does not necessarily measure weather resistance.

Dimensions and Permissible Variations

4. (*a*) The average net area of the unit shall not be less than 75 per cent of the gross area.

(*b*) No over-all dimension shall vary more than 3 per cent over or under the specified dimension for any form of unit.

Visual Inspection

5. (*a*) All units shall be sound and free from cracks or other defects that would interfere with the proper placing of the unit, or impair the strength or permanence of the construction.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-15 on Manufactured Masonry Units.

[2] Prior to adoption as standard, these specifications were published as tentative from 1939 to 1940.

[3] When fire tests are required they shall be conducted in accordance with the Standard Methods of Fire Tests of Building Construction and Materials (A.S.T.M. Designation: C 19), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(*b*) Units that are intended to serve as a base for plaster or stucco shall have a sufficiently rough surface to afford good bond.

Marking

6. All units shall bear a distinctive mark of the manufacturer or shall be otherwise readily identified as to origin.

Sampling and Testing

7. (*a*) The purchaser or his authorized representative shall be accorded proper facilities to inspect and sample the units at the place of manufacture from the lots ready for delivery. At least ten days should be allowed for completion of the tests.

(*b*) Units shall be sampled and tested in accordance with the Standard Methods of Sampling and Testing Concrete Masonry Units (A.S.T.M. Designation: C 140) of the American Society for Testing Materials.[4]

Rejection

8. In case the shipment fails to conform to the requirements, the manufacturer may sort it, and new specimens shall be selected by the purchaser from the retained lot and tested at the expense of the manufacturer. In case the second set of specimens fails to conform to the test requirements, the entire lot shall be rejected.

Expense of Tests

9. Except as specified in Section 8, and unless otherwise agreed, the expense of inspection and testing shall be borne by the purchaser.

[4] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

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Standard Specifications for

CONCRETE MASONRY UNITS FOR CONSTRUCTION OF CATCH BASINS AND MANHOLES[1]

A.S.T.M. Designation: C 139 - 39

ADOPTED, 1939.[2]

Reapproved in 1944 Without Change.

This Standard of the American Society for Testing Materials is issued under the fixed designation C 139; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover solid, precast, segmental concrete masonry units made from portland cement and suitable aggregates such as sand, gravel, crushed stone, and air-cooled blast-furnace slag. The units are intended for use in the construction of catch basins and manholes.

Physical Requirements

2. At the time of delivery to the site of the work the units shall conform to the physical requirements prescribed in Table I.

TABLE I.—PHYSICAL REQUIREMENTS.

Compressive Strength, min., psi. (Average Gross Area)		Water Absorption, max. per cent	
Average of 5 Units	Individual Unit	Average of 5 Units	Individual Unit
2500	2000	8	10

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-15 on Manufactured Masonry Units.

[2] Prior to adoption as standard, these specifications were published as tentative from 1938 to 1939.

Dimensions and Permissible Variations

3. (*a*) The thickness of unit shall be not less than 5 in.

(*b*) No over-all dimension shall vary more than 3 per cent over or under the specified dimension for any form of unit.

Visual Inspection

4. All units shall be sound and free from cracks or other defects that would interfere with the proper placing of the unit.

Marking

5. All units shall bear a distinctive mark of the manufacturer or shall be otherwise readily identified as to origin.

Sampling and Testing

6. (*a*) The purchaser of his authorized representative shall be accorded proper facilities to inspect and sample the units at the place of manufacture from the lots ready for delivery. At least ten days should be allowed for completion of the tests.

(*b*) Units shall be sampled and tested in accordance with the Standard Meth-

ods of Sampling and Testing Concrete Masonry Units (A.S.T.M. Designation: C 140) of the American Society for Testing Materials.[3]

Rejection

7. In case the shipment fails to conform to the requirements, the manufacturer may sort it, and new specimens shall be selected by the purchaser from the retained lot and tested at the expense of the manufacturer. In case the second set of specimens fails to conform to the test requirements, the entire lot shall be rejected.

Expense of Tests

8. Except as specified in Section 7, and unless otherwise agreed, the expense of inspection and testing shall be borne by the purchaser.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: A84.1-1942

Standard Methods of

SAMPLING AND TESTING CONCRETE MASONRY UNITS[1]

A.S.T.M. Designation: C 140 - 39

ADOPTED, 1939.[2]

Reapproved in 1944 Without Change.

This Standard of the American Society for Testing Materials is issued under the fixed designation C 140; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods cover procedures for the sampling and testing of concrete masonry units for compressive strength, absorption, weight, and moisture content.

NOTE.—Procedures for the sampling and testing of concrete brick are covered in the Standard Methods of Sampling and Testing Brick (A.S.T.M. Designation: C 67) of the American Society for Testing Materials.[3]

Definitions of Terms

2. The Standard Definitions of Terms Relating to Methods of Testing (A.S.T.M. Designation: E 6) of the American Society for Testing Materials[3] shall be considered as applying to the terms used in these methods.

SAMPLING

Selection of Test Specimens

3. For purpose of tests, full-size concrete masonry units shall be selected by the purchaser or by his authorized representative. Specimens shall be representative of the whole lot of units from which they are selected. If test specimens are selected at the site of the work, units for moisture content tests shall be taken within 48 hr. after delivery.

Number of Specimens

4. For the strength, absorption and moisture content determinations, 10 individual units shall be selected from each lot of 10,000 units or fraction thereof and 20 individual units from each lot of more than 10,000 and less than 100,000 units. For lots of more than 100,000 units 10 individual units shall be selected from each 50,000 units or fraction thereof contained in the lot. In no case shall less than 10 units be taken. Additional specimens may be taken at the discretion of the purchaser.

Identification

5. (*a*) Each specimen shall be marked so that it may be identified at any time. Markings shall not cover more than 5 per cent of the superficial area of the specimen.

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee C-15 on Manufactured Masonry Units.

[2] Prior to adoption as standard, these methods were published as tentative from 1938 to 1939, being revised in 1939.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(*b*) Units for moisture content tests shall be weighed immediately after sampling and marking.

COMPRESSIVE STRENGTH

Test Specimens

6. Five air-dry full-size units shall be tested.

Capping Test Specimens

7. (*a*) A quantity of plastic capping mortar, made of one part (by volume) of portland cement and one part of calcined gypsum (plaster of Paris) mixed with sufficient water to spread evenly, shall be placed on a plane surface which has been coated with oil. The surface to be capped shall be placed on this mortar, and the specimen, so held that its axis is at right angles to the capping surface, shall be firmly pressed down with a single motion.

(*b*) The average thickness of the cap after the extruded capping mortar has been removed and the edges trued shall be not more than $\frac{1}{8}$ in. Patching of caps after setting shall not be permitted. Imperfect caps shall be removed and replaced with new ones.

(*c*) The caps shall be aged at least 6 hr. before the specimens are tested.

Procedure

8. (*a*) *Position of Specimens.*—All specimens shall be tested in a position such that the load is applied in the same direction as in service.

(*b*) *Testing Machine.*—The testing machine shall conform to the requirements of the Standard Methods of Verification of Testing Machines (A.S.T.M. Designation: E 4) of the American Society for Testing Materials.[3]

(*c*) *Bearing Blocks.*—The upper bearing shall be a spherically seated, hardened metal block firmly attached at the center of the upper head of the machine. The center of the sphere shall lie at the center of the surface of the block in contact with the specimen. The block shall be closely held in its spherical seat, but shall be free to turn in any direction. The diameter of the bearing surface shall be at least 6 in. A hardened metal bearing block shall be used beneath the specimen to minimize wear of the lower platen of the machine. The bearing block surfaces intended for contact with the specimen should have a hardness not less than C60 Rockwell number (Brinell number 620). These surfaces shall not depart from plane surfaces by more than 0.001 in. When the bearing area of the spherical bearing block is not sufficient to cover the area of the specimen, a steel plate with surfaces machined to true planes within plus or minus 0.001 in., and with a thickness equal to at least one-third of the distance from the edge of the spherical bearing to the most distant corner, shall be placed between the spherical bearing block and the capped specimen.

(*d*) *Speed of Testing.*—The speed of the moving head of the testing machine shall be not more than 0.05 in. per min.

Calculations and Report

9. (*a*) The compressive strength of a concrete masonry unit shall be taken as the maximum load in pounds divided by the gross cross-sectional area of the unit in square inches. The gross area of a unit is the total area of a section perpendicular to the direction of the load, including areas within cells and within re-entrant spaces unless these spaces are to be occupied in the masonry by portions of adjacent masonry.

(*b*) *Net Area.*—The average percentage of net area of the unit is equal to the

net volume divided by the gross volume and multiplied by 100:

$$\text{Net area, per cent} = \frac{\text{net vol.} \times 100}{\text{gross vol.}}$$

$$\text{Net volume, cu. ft.} = \frac{\text{dry wt. of unit}}{\text{wt. per cu. ft.}}$$

$$\text{Gross volume, cu. ft.} = \frac{T \times H \times L}{1728}$$

$$\text{Weight per cu. ft., lb.} = \frac{\text{dry wt.} \times 62.4}{\text{wet wt.} - \text{suspended immersion wt.}}$$

where:

T = thickness of the unit in inches,
H = height of the unit in inches, and
L = length of the unit in inches.

(*c*) The results shall be reported separately for each unit, with the average for the five units.

ABSORPTION

Test Specimens

10. Five full-size units shall be used.

Accuracy of Weighings

11. The balance used shall be sensitive to within 0.5 per cent of the weight of the smallest specimen tested.

Procedure

12. (*a*) *Saturation.*—The test specimens shall be completely immersed in water at room temperature at from 15.6 to 26.7 C. (60 to 80 F.) for 24 hr. The specimens shall then be weighed while suspended with a metal wire and completely submerged in water. They shall be removed from the water and allowed to drain for 1 min. by placing on a $\frac{3}{8}$-in. or coarser wire mesh, visible surface water being removed with a damp cloth, and immediately weighed.

(*b*) *Drying.*—Subsequent to saturation all specimens shall be dried in a drier or oven at 100 to 115 C. (212 to 239 F.) and weighed at 24-hr. intervals until the loss in weight does not exceed 1 per cent in a 24-hr. period.

Calculations and Report

13. (*a*) *Absorption.*—The absorption in pounds per cubic foot of concrete is equal to 62.4 times the difference between the wet and dry weights divided by the difference between the wet weight and the suspended weight immersed:

$$\text{Absorption, lb. per cu. ft.} = \frac{(\text{wet wt.} - \text{dry wt.}) \times 62.4}{(\text{wet wt.} - \text{suspended wt. immersed})}$$

The absorption expressed as a percentage of the dry weight is equal to the difference between the wet and dry weights divided by the dry weight and multiplied by 100:

$$\text{Absorption, per cent} = \frac{(\text{wet wt.} - \text{dry wt.}) \times 100}{\text{dry wt.}}$$

(*b*) *Moisture Content.*—The moisture content of the units at time of sampling (expressed as a percentage of the total absorption) is the difference between the average weight as sampled and the average dry weight divided by the difference between the average wet weight and the average dry weight and multiplied by 100:

$$\text{Moisture content, per cent} = \frac{(\text{sampled wt.} - \text{dry wt.}) \times 100}{(\text{wet wt.} - \text{dry wt.})}$$

(*c*) *Report.*—The results shall be reported separately for each unit, with the average for the five units.

Standard Methods of Test for

ABSORPTION AND APPARENT SPECIFIC GRAVITY OF NATURAL BUILDING STONE[1]

A.S.T.M. Designation: C 97 - 36

ADOPTED, 1936.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 97; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods of test cover the procedures for determining the absorption by weight or by volume, and the apparent specific gravity of natural building stones used for exterior or interior construction or decorative purposes. The calculation of the dry weight of the stone in pounds per cubic foot may also be made from the results of the apparent specific gravity test.

ABSORPTION

Test Specimens

2. Samples shall be supplied of sufficient size to provide not less than three test specimens of some regular form such as a cube, prism, or cylinder whose greatest dimension shall be not more than 3 in. and whose least dimension shall be not less than 2 in. All surfaces of specimens shall be smooth finished. No chisels or other hand tools shall be used in finishing specimens.

Procedure

3. (*a*) The specimens shall be dried in an oven at 110 to 120 C. for at least 24 hr.

(*b*) After drying, the specimens may be cooled in the room for 30 min. and then weighed. When the specimens cannot be weighed immediately after cooling, they shall be placed in a desiccator. The weight shall be determined to the nearest 0.05 g.

(*c*) The specimens shall be entirely immersed in distilled water at approximately 20 C. for two weeks. At the end of this period the specimens shall be removed one at a time, all faces and edges thoroughly wiped off with a damp cloth, and each specimen immediately weighed to the nearest 0.05 g.

NOTE.—The operator should distinguish between a damp cloth and a wet one. In starting the operation, the towel should be sprinkled lightly. When the surface of the specimen is properly wiped off, it should appear dry.

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee C-18 on Natural Building Stones and Slate.

[2] Prior to adoption as standard, these methods were published as tentative from 1930 to 1936, being revised in 1931. Editorially revised and rearranged in 1939.

Calculations and Report

4. (*a*) The percentage of absorption shall be calculated as follows:

$$\text{Absorption, per cent} = \frac{B - A}{A} \times 100$$

where:

A = weight of the dried specimen, and

B = weight of the specimen after immersion.

(*b*) In general, the average of all the tests shall be reported as the absorption of the stone. Individual determinations which differ by more than 25 per cent from the average of all the determinations may be disregarded when there is evidence that such results are of uncertain value. The absorption shall then be reported as the average of the remaining determinations, but all determinations shall be reported as information.

(*c*) The absorption value obtained from this test is the percentage absorption by weight. To convert the result to percentage absorption by volume, multiply by the value of the apparent specific gravity as determined with the same specimens in accordance with Sections 5 and 6.

(*d*) The report of the absorption test shall state whether the absorption has been determined according to weight or volume and, if the percentage of volume absorption is reported, there shall also be reported the percentage absorption by weight and the apparent specific gravity.

APPARENT SPECIFIC GRAVITY

Weighing

5. To obtain the apparent specific gravity, the weight of the specimen suspended in water shall be determined immediately after the weighing of the specimen described in Section 3.

NOTE.—When weighing the specimen suspended in water, various methods of suspending the specimen from the balance beam are in use. Fine silk thread or a cradle of light wire whose weight has been carefully determined may be convenient. The cradle should be of such construction that there will be no possibility of air bubbles forming in it. The water container should be of glass and may be supported on a platform which straddles the balance pan, this method allowing the weighings to be made without detaching the pan.

Calculations and Report

6. (*a*) The apparent specific gravity shall be calculated as follows:

$$\text{Apparent specific gravity} = \frac{A}{B - C}$$

where:

A = weight of the dried specimen,

B = weight of the wet specimen after immersion, and

C = weight of the specimen suspended in water.

(*b*) The apparent specific gravity shall be calculated for those specimens whose absorption values are to be averaged as described in Section 4 (*d*), and the average of the apparent specific gravities so calculated shall be reported as that of the stone.

NOTE.—To determine the weight of the dry stone in pounds per cubic foot, multiply the apparent specific gravity by 62.4.

Standard Method of

FLEXURE TESTING OF NATURAL BUILDING STONE[1]

DETERMINATION OF MODULUS OF RUPTURE

A.S.T.M. Designation: C 99 - 36

ADOPTED, 1936.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 99: the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test covers the procedure for determining the modulus of rupture of natural building stones used for exterior or interior construction or decorative purposes.

Apparatus

2. (*a*) *Testing Machine.*—The testing machine shall be sensitive to 5 lb. when loaded to 500 lb.

(*b*) *Knife-Edges.*—For the support of the test specimen a set of knife-edges similar to those shown in Fig. 1 shall be provided. The load shall be applied through a third knife-edge of the same type.

Test Specimens

3. (*a*) Samples shall be representative of the material and shall consist of sections cut from apparently sound material, preferably quarry blocks or slabs. The direction of bedding or rift planes shall be plainly marked on the sample. The sample supplied shall be of sufficient size to provide all of the specimens required for the test.

(*b*) The test specimens shall be sawed from the sample and shall be finished as closely as practicable to 12 by 4 by 1 in. in size either by the use of grinding wheels or by hand rubbing with abrasive powders on steel plates. The 12 by 4-in. faces shall be made as nearly plane and parallel as practicable. No hand cutting with chisels or other tools shall be permitted.

Types of Specimens

4. Three or more specimens shall be prepared for each required combination of load application and bedding or rift plane direction, such as are shown in Fig. 2 and noted below:

(*1*) Load applied perpendicular to bedding or rift planes,

(*2*) Load applied parallel to bedding or rift planes, and

(*3*) Load applied perpendicular to

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-18 on Natural Building Stones and Slate.

[2] Prior to adoption as standard, this method was published as tentative from 1930 to 1936, being revised in 1931. Editorially revised and rearranged in 1939.

bedding or rift planes with the bedding or rift planes vertical.

Marking Specimens

5. The load-bearing surfaces and the direction of bedding or rift planes shall be carefully marked on each specimen after finishing.

Measuring Specimens

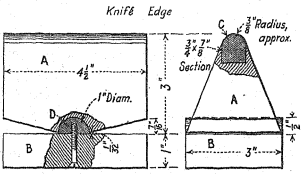
6. The thickness and width of the specimen at the mid-section shall be measured to the nearest 0.01 in.

Conditioning

7. Before testing, the specimens shall be dried in an oven at 110 to 120 C. for 24 hr.

Procedure

8. The test specimen shall be supported on the knife-edges on 10-in. centers and the load applied at the center of the specimen through the third knife-edge (see Fig. 2). The load shall be applied at the rate of 100 lb. per min. The breaking load shall be recorded to the nearest 5 lb.



A, B = Cast Iron Wedges.
C, D = Cold-Rolled Steel.

FIG. 1.—Self-Adjusting Knife-Edge Bearing and Load-Application Device.

Calculations and Report

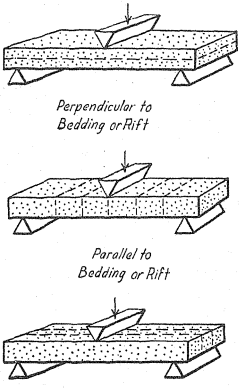
9. (*a*) The modulus of rupture shall be calculated as follows:

$$R = \frac{3wl}{2bd^2}$$

where:

R = modulus of rupture,
w = breaking load in pounds,
l = length of span in inches,
b = width of specimen in inches, and
d = thickness of specimen in inches.

(*b*) The average value of the modulus obtained from a group of not less than two specimens shall be reported as the modulus of rupture for the condition of loading and direction of bedding or rift used. In case any specimen shows a value 25 per cent lower than the average of its group, it shall be examined for defects, and if the low value appears to be due to imperfections, the average of the other specimens in the group shall be reported as the modulus of rupture of the group. All determinations shall be reported as information.



Dashed lines indicate direction of bedding or rift. Arrows indicate direction of loading.

FIG. 2.—Methods of Applying Load with Reference to Bedding or Rift.

Standard Method of Test for

MODULUS OF ELASTICITY OF NATURAL BUILDING STONE[1]

A.S.T.M. Designation: C 100 - 36

ADOPTED, 1936.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 100; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test covers the procedure for determining the modulus of elasticity in compression or in flexure of natural building stones used for exterior or interior construction or decorative purposes.

Apparatus

2. (*a*) *Elasticity in Flexure.*—For determining the elasticity in flexure, the testing machine and knife-edges shall conform to the requirements prescribed in Section 2 of the Standard Method of Flexure Testing of Natural Building Stone (Determination of Modulus of Rupture) (A.S.T.M. Designation: C 99) of the American Society for Testing Materials.[3] A suitable deflectometer capable of measuring the deflection of the specimen to 0.0001 in. and of such design that it may be attached to the specimen shall be used. A measuring device of satisfactory type is shown in Fig. 1 (*b*).

(*b*) *Elasticity in Compression.*—For determining the elasticity in compression, any form of standard testing machine may be used for the compression test together with a spherical bearing block large enough to entirely cover the specimen, but not greater in diameter than three times the width of the loaded face of the specimen. A compressometer of the averaging type capable of measuring the compression of the specimen to 0.00002 in. shall be used. A measuring device of satisfactory type is shown in Fig. 1 (*a*).

Test Specimens

3. (*a*) Samples shall be representative of the material and shall consist of sections cut from apparently sound material, preferably quarry blocks or slabs. The direction of the bedding planes or rift shall be plainly marked on the sample. The sample shall be of sufficient size to allow the preparation of not less than three specimens having the bedding planes or rift running in

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-18 on Natural Building Stones and Slate.

[2] Prior to adoption as standard, this method was published as tentative from 1931 to 1936. Editorially revised and rearranged in 1939.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

the proper direction for the kind of test specified, that is, either parallel or perpendicular to the face of the specimen to which pressure is to be applied in the test.

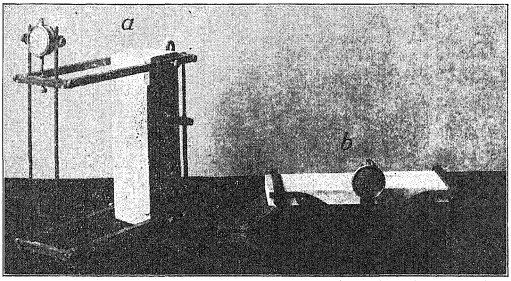
(*b*) *Elasticity in Flexure.*—For determining elasticity in flexure, the same size and shape specimens specified in Sections 3 and 4 of the Standard Method of Flexure Testing of Natural Building Stone (Determination of Modulus of Rupture) (A.S.T.M. Designation: C 99) of the American Society for Testing Materials[3] shall be used.

Marking Specimens

4. The load-bearing surfaces and the direction of the bedding planes or rift shall be carefully marked on each specimen after finishing.

Measuring Specimens

5. The dimensions of the specimen shall be measured to the nearest 0.01 in. The width and thickness at the center section shall be used in calculating the elasticity in flexure and also in compression. The length or height of the specimen shall be recorded and the gage length used shall be measured and centered along the length or height.



(*a*) Compressometer. (*b*) Deflectometer.

FIG. 1.—Assembled Views of Apparatus for Modulus of Elasticity Test.

(*c*) *Elasticity in Compression.*—For determining elasticity in compression, prismatic pieces 3½ by 3½ by 12 in. shall be used, and the sides of the blocks shall be finished to approximate planes.

(*d*) The specimens shall be cut from the original samples with saws. No hand cutting with chisels or other tools shall be permitted. The load-bearing surfaces shall be finished to as nearly true and parallel planes as possible, grinding wheels or the use of abrasive powders and rubbing plates affording convenient means of obtaining satisfactory surfaces.

Conditioning

6. (*a*) *Dry Condition.*—Specimens to be tested in the dry condition shall be dried in an oven at 110 to 120 C. for 24 hr. immediately preceding the test.

(*b*) *Wet Condition.*—Specimens to be tested in the wet condition shall be completely immersed in water at room temperature for at least two weeks, and shall be kept in water until immediately preceding the test.

Procedure

7. (*a*) *Elasticity in Flexure.*—For determining elasticity in flexure, the slab shall be set up on the knife-edge bearings, the deflectometer attached, and an initial load of 20 lb. applied. After setting the dial to approximately zero and noting the readings, the load shall be increased by 10-lb. increments and readings of the deflectometer made for each 50 lb. of load. The reading of the dial shall be recorded to the nearest 0.0001 in. The loading shall be continued until failure of the specimen occurs.

(*b*) *Elasticity in Compression.*—For determining elasticity in compression, the compressometer shall be attached to the specimen and then the specimen accurately centered in the testing machine with the spherical bearing block on the top of the specimen. The center of the spherical surface of the bearing block shall lie in the face of the bearing block in contact with the top bearing surface of the specimen. The spherical bearing shall be kept well lubricated to insure accurate adjustment, which should be made by hand under a small initial load. The speed of the moving head of the testing machine during the application of the load shall be not more than 0.05 in. per min.

An initial load of 500 lb. shall be applied and the dial set to zero. Load increments of 5000 lb. each shall be applied thereafter up to approximately one-half of the estimated breaking load of the specimen, stopping the testing machine at each 5000-lb. loading to read the compressometer, the readings being recorded to the nearest 0.00002 in. After taking the reading at the last 5000-lb. load increment, the load shall be gradually released to 500 lb. and the dial again read. The dial shall then be reset to zero if necessary and a second application of the load made as before. The load shall be fully removed after the reading at 500 lb. following the second load application and the specimen removed from the testing machine. The compressometer shall then be removed and reattached with the specimen turned 90 deg. from its original position. A third application of the load shall then be made as before.

Calculations

8. (*a*) *Elasticity in Flexure.*—The modulus of elasticity in flexure shall be calculated by use of a load-deflection diagram on which the deflections shall be plotted against the loads. A straight line shall be drawn through the origin parallel to the proportional part of the curve. The modulus of elasticity shall then be calculated as follows:

$$E = \frac{wl^3}{4\Delta bd^3}$$

where:

E = modulus of elasticity in pounds per square inch,
w = load ordinate at some convenient point on the line through the origin,
Δ = deformation ordinate of load point chosen,
l = length between supporting knife-edges in inches,
b = width of specimen at the center in inches, and
d = thickness of specimen at the center in inches.

(*b*) *Elasticity in Compression.*—The modulus of elasticity in compression shall be calculated by use of a load-deformation diagram on which the deformations shall be plotted against the loads. A straight line shall be drawn through the origin parallel to the proportional part of the curve. The

modulus of elasticity shall be calculated as follows:

$$E = \frac{s}{d} = \frac{P}{A\,d}$$

where:

E = modulus of elasticity in pounds per square inch,
s = unit stress in pounds per square inch,
d = unit deformation at some convenient point on the line through the origin,
P = total load on specimen at point corresponding to d on line through the origin, and
A = area of specimen at center section.

Report

9. The report shall include the following:

(*a*) The individual and averaged modulus of elasticity in pounds per square inch in flexure or compression for each group of specimens. Each individual test shall be reported, and for the modulus of elasticity of the material the average of all tests shall be used except where an individual result varies from the average of its group by more than 25 per cent, in which case such individual result shall be reported as information only.

(*b*) The types and approximate sizes of specimens used,

(*c*) The condition of the specimen when tested, whether wet or dry,

(*d*) The direction of the application of the load relative to the bedding planes or rift of the specimens. When the load has been applied on a line perpendicular to the bedding or rift planes, the result shall be reported as "modulus of elasticity in pounds per square inch perpendicular to the bedding plane or rift." When the load has been applied on a line parallel to the bedding or rift planes, the result shall be reported as "modulus of elasticity in pounds per square inch parallel to the bedding plane or rift."

(*e*) A definite statement as to the manner in which the specimens were prepared and finished, and

(*f*) The type of testing machine used.

Standard Method of

SHEAR TESTING OF NATURAL BUILDING STONE[1]

A.S.T.M. Designation: C 102 - 36

ADOPTED, 1936.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 102; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test covers the procedure for determining the shearing strength of natural building stones used for exterior or interior construction or decorative purposes.

Apparatus

2. Either the modified Johnson shear tool or the Dutton punching shear device may be used in conjunction with any standard testing machine of sufficient capacity. A spherical bearing block of sufficient size to cover the upper end of the plunger of the punching shear device shall be used if that apparatus is employed. Details of the two types of apparatus are shown in Figs. 1 and 2, and assembled views with specimens in place are shown in Fig. 3.

Test Specimens

3. (*a*) Samples shall be representative of the material and shall consist of sections cut from apparently sound material, preferably quarry blocks or slabs. The direction of the bedding planes or rift shall be plainly marked on the samples. The sample shall be of sufficient size to allow the preparation of not less than three specimens having the bedding planes or rift running in the proper direction for the kind of test specified, that is, either parallel or perpendicular to the face of the specimen to which pressure is to be applied in the test.

(*b*) Specimens for use in the Johnson shear tool shall be bars 2 by 2 in. in section and not less than 7 in. in length. For the Dutton punching shear device, slabs 1 in. in thickness, 4 in. in width, and not less than 4 in. in length shall be used.

NOTE.—The most convenient form of specimen for the punching shear test is the flat slab obtained when the flexure test is made, the broken pieces of the slab used in the flexure test nearly always being of the proper length while the slab itself was previously cut to 1 in. in thickness and 4 in. in width. The use of these slabs from the flexure test will eliminate the separate preparation of shear test specimens.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-18 on Natural Building Stones and Slate.

[2] Prior to adoption as standard, this method was published as tentative from 1931 to 1936. Editorially revised in 1939 and 1942.

(*c*) The specimens shall be cut from the original samples with saws. No hand cutting with chisels or other tools shall be permitted. The load-bearing surfaces shall be finished to as nearly true and parallel planes as possible, grinding wheels or the use of abrasive powders and rubbing plates affording convenient means of obtaining satisfactory surfaces.

the specimen shall be included in the report as information only.

(*b*) When using the Dutton punching device, center lines shall be laid off on one surface of the slab and the thickness of the slab measured to the nearest 0.01 in. at not less than three points approximately equidistant around the circumference of a 2-in. circle centered on the intersection of the two

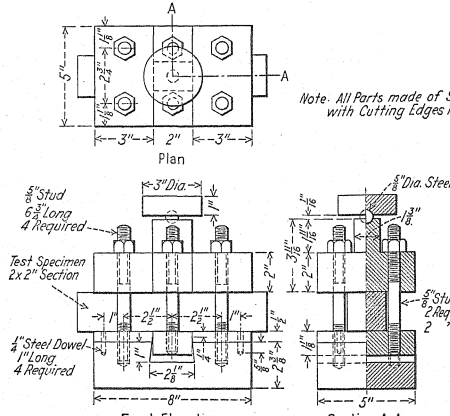


FIG. 1.—Details of Modified Johnson Shear Tool.

Marking Specimens

4. The load-bearing surfaces and the direction of the bedding planes or rift shall be carefully marked on each specimen after finishing.

Measuring Specimens

5. (*a*) For tests with the Johnson shear apparatus the specimen shall be measured to the nearest 0.01 in. at the center section. The area under shearing stress shall be taken as twice the area of this center section. The length of center lines. The width and length of the specimen shall be included in the report as information only.

Conditioning

6. (*a*) *Dry Condition.*—Specimens to be tested in the dry condition shall be dried in an oven at 110 to 120 C. for 24 hr. immediately preceding the test.

(*b*) *Wet Condition.*—Specimens to be tested in the wet condition shall be completely immersed in water at room temperature for at least two weeks,

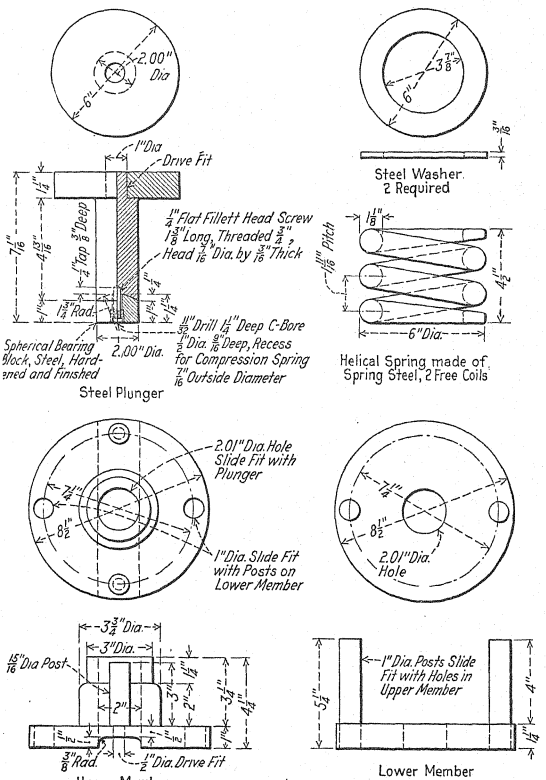


FIG. 2.—Details of Dutton Punching Shear Device.

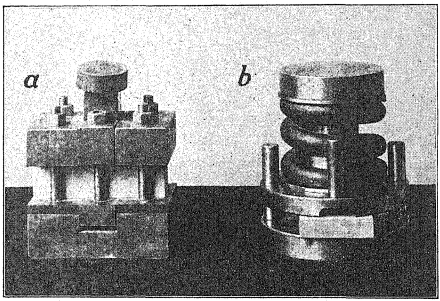
and shall be kept in water until immediately preceding the test.

(*c*) If the broken slabs from the flexure test are to be used, they shall be kept in a desiccator until tested if not tested in shear immediately upon completion of the flexure test.

Procedure

7. (*a*) *Using the Johnson Shear Tool.*—The specimen shall be carefully centered in the shear tool as shown in Fig. 1, and the bolts drawn up tightly. The tool shall then be centered in the testing machine with the center of the spherical block in contact with the center of the top portion of the plunger of the shear tool. The spherical bearing shall be kept well lubricated to insure accurate adjustment which should be made by hand under a small initial load. The speed of the moving head of the testing machine during the application of the load shall be not more than 0.05 in. per min. During the test, the beam of the testing machine shall be kept constantly in a floating position.

(*b*) *Using the Dutton Punching Shear Device.*—The specimen shall be carefully centered between the upper and lower plates of the punching device so that the measured section of the specimen is under the plunger. The upper plate shall be carefully lowered to contact with the specimen. The punching device shall then be centered in the testing machine with the center of the spherical bearing block in contact with the center of the top portion of the plunger of the shear device. The spherical bearing shall be kept well lubricated to insure accurate adjustment which should be made by hand under a small initial load. The speed of the moving head of the testing machine during the application of the load shall be not more than 0.05 in. per min. During the test the beam of the testing machine shall be kept constantly in a floating position.



(*a*) Modified Johnson Shear Tool. (*b*) Dutton Punching Shear Device.
FIG. 3.—Assembled Views of Shear Test Apparatus.

Calculations

8. (*a*) *Using Johnson Shear Tool.*—The shearing strength shall be calculated as follows:

$$S = \frac{W}{2A}$$

where:

S = shearing strength in pounds per square inch,
W = total maximum load in pounds indicated by the testing machine, and
A = area in square inches of the center section of the specimen.

(*b*) *Using the Dutton Punching Device.*—The shearing strength shall be calculated as follows:

$$S = \frac{W_T - W_I}{\pi d T}$$

where:

S = shearing strength in pounds per square inch,
W_T = total maximum load in pounds indicated by the testing machine,
W_I = initial load in pounds required to bring the plunger in contact with the surface of the specimen,
d = diameter in inches of the plunger, and
T = thickness in inches of the specimen.

Report

9. The report shall include the following:

(*a*) The individual and averaged shearing strengths in pounds per square inch for each group of specimens. Each individual test shall be reported, and for the shearing strength of the material the average of all tests shall be used except where an individual result varies from the average of its group by more than 25 per cent, in which case that individual result shall be reported as information only.

(*b*) The type and approximate sizes of specimens used,

(*c*) The condition of the specimen when tested, whether wet or dry,

(*d*) The direction of the application of the load relative to the bedding planes or rift of the specimens. When the load has been applied on a line perpendicular to the bedding or rift planes, the result shall be reported as "shearing strength in pounds per square inch perpendicular to the bedding plane or rift." When the load has been applied on a line parallel to the bedding or rift planes, the result shall be reported as "shearing strength in pounds per square inch parallel to the bedding plane or rift."

(*e*) A definite statement as to the manner in which the specimens were prepared and finished, and

(*f*) The type of shear appliance used.

Standard Methods of

FLEXURE TESTING OF SLATE[1]

MODULUS OF RUPTURE, MODULUS OF ELASTICITY

A.S.T.M. Designation: C 120 – 31

ADOPTED, 1931.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 120; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Due to the unique properties of slate the flexure test is better adapted to use for strength and elasticity determinations than either compression or tension tests. Furthermore, several uses of slate are such that these determinations are of special interest and value, besides furnishing comparative data.

The property of slate termed "grain" causes a slab of the material to break transversely in one direction somewhat more readily than at right angles to this direction. For this reason it is desirable to test the strength and elasticity both parallel and perpendicular to the grain.

In the quarrying of slate, blasting is frequently resorted to and for this reason certain portions of the material may have been unduly strained. Low or erratic strength results on some of the test specimens should be regarded in the light of defective material.

Scope

1. These methods of test cover the procedures for determining the modulus of rupture and modulus of elasticity of slate by means of flexure tests.

MODULUS OF RUPTURE

Test Specimens

2. (*a*) *Structural or Electrical Slate.*—Six representative specimens, 12 by 1½ by 1 in. in size, of the particular slate under consideration shall be tested.

(*b*) *Roofing Slate.*—At least six specimens 4 in. in width, 12 in. or more in length, and of thickness equal to that of the slate shingle shall be tested.

Preparation of Specimens

3. (*a*) *Structural or Electrical Slate.*—The slate for the test shall have been split to a thickness of approximately 1¼ in. and then sawed into strips 12 in. in length by 1½ in. in width. Half of these shall be cut with the length parallel to the grain and half with the length perpendicular to the grain (Note 1). The 12 by 1½-in. faces shall then be planed or rubbed down to a thickness of ap-

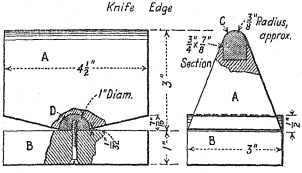
[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee C-18 on Natural Building Stones and Slate.

[2] Prior to adoption as standard, these methods were published as tentative from 1925 to 1931, being revised in 1927 and 1931. Editorially revised and rearranged in 1939.

proximately 1 in., care being taken to have the finished surfaces as nearly parallel as practicable.

(*b*) *Roofing Slate.*—The specimens shall be sawed from the shingles with the long dimension parallel to the long dimension of the shingle (Note 2) so that no saw cut is nearer than 1 in. to the sheared edge of the shingle. Split faces shall not be refinished.

NOTE 1.—When it is desired to test a finished slab of slate intended for structural or electrical uses, the specimens may be sawed from the sides. Those cut the long way of the slab may be considered to be parallel to the grain and those cut the short way, perpendicular to the grain.



A, B = *Cast Iron Wedges.*
C, D = *Cold-Rolled Steel.*

FIG. 1.—Self-Adjusting Knife-Edge Bearing and Load-Application Device.

NOTE 2.—This gives a specimen parallel to the grain and when broken transversely, the fracture will occur across the grain. Ordinarily, it is not considered necessary to test roofing slate in both directions of the grain.

Conditioning

4. Before testing, the specimens shall be dried in an oven at 110 ± 3 C. for 24 hr.

Measuring Specimens

5. The thickness at the middle shall be measured to the nearest 0.001 in., and the width shall be measured to the nearest 0.01 in.

Procedure

6. The testing machine shall be sensitive to 5 lb. when loaded to 500 lb. The specimen shall be supported flatwise on knife-edges of the type shown in Fig. 1 spaced 10 in. on centers. The load shall be applied through a third knife-edge at the middle of the span at a rate of approximately 100 lb. per min. The breaking load shall be recorded to the nearest 5 lb.

NOTE.—The following method of marking the specimens before testing is recommended:

By means of a try square the middle section and two end sections, 5 in. distant from the center, may be circumscribed. These marks serve as a guide to the operator in properly centering and aligning the specimen in the testing machine.

Calculations

7. The modulus of rupture shall be calculated as follows:

$$R = \frac{3wl}{2bd^2}$$

where:

R = modulus of rupture in pounds per square inch,
w = breaking load in pounds,
l = span length between supporting knife-edges,
b = width of specimen at the center in inches, and
d = thickness of specimen at the center in inches.

Report

8. The average of the specimens cut parallel to the grain from structural or electrical slate, or parallel to the long dimension of the shingle in the case of roofing slate, shall be reported as the modulus of rupture "across the grain." The average of the specimens cut perpendicular to the grain shall be reported as the modulus of rupture "with the grain." In case any specimen shows a value 25 per cent lower than the average of its group, it shall be examined for defects, and if the low value appears to

be due to an imperfection, the average of the other five shall be reported as the modulus of rupture of the group. All determinations shall be reported as information.

MODULUS OF ELASTICITY

Test Specimens

9. The test for modulus of elasticity, when required, is performed in conjunction with the modulus of rupture test described in Sections 2 to 8, and hence the test specimens are of the same type and the measuring and drying operations are the same as specified in Sections 2 to 5.

Procedure

10. The specimen shall be supported and loaded in the same way as for the modulus of rupture determination except that the loading shall be interrupted at suitable intervals for deflection measurements. A deflectometer capable of measuring accurately to 0.001 in. shall be used to obtain the deflections at the middle of the span. For test specimens of structural or electrical slate it will be satisfactory to record the deflections for each 50-lb. increment of load, but for test specimens of roofing slate the deflections shall be recorded for each 20-lb. increment.

NOTE.—It is not ordinarily feasible to set the deflectometer to read zero when there is no load on the specimen. The best practice is to put a small initial load on the specimen, as 10 lb. and set the deflectometer to read zero for this load. Since it is only the slope of the stress-strain curve that is desired, this initial load does not affect the final result.

Calculations

11. The load-deflection readings shall be plotted on cross-section paper to a convenient scale and a straight line drawn to represent, as nearly as possible, the average of the plotted points.[3] If the line does not pass through the zero point, a corrected line shall be drawn through this point parallel to the stress-strain line. The modulus of elasticity, E, shall be calculated from the coordinates of some convenient point on the corrected line by means of the following formula:

$$E = \frac{W'l^3}{4\Delta bd^3}$$

where:

W' = load coordinate of the point,
Δ = deformation ordinate of the point,
l = length of span in inches,
b = width of specimen at the center in inches, and
d = thickness of specimen at the center in inches.

Report

12. In general, the average of the results obtained for specimens prepared with the length parallel to the grain shall be reported as the modulus of elasticity across the grain. Likewise, the average of the results obtained on specimens cut with the length perpendicular to the grain shall be reported as the modulus of elasticity with the grain. In case an individual determination differs by more than 25 per cent from the average of its group, it may be disregarded if there is evidence that the determination was of uncertain value, but all determinations shall be reported as information.

[3] Slate does not show a definite yield point in the stress-strain curve.

Standard Method of Test for

WATER ABSORPTION OF SLATE[1]

A.S.T.M. Designation: C 121 - 31

ADOPTED, 1931.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 121; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

It is often desirable to gain some idea of the porosity of a material in question. Actual determinations of the pore space require the use of rather elaborate and refined equipment as well as considerable precision in carrying out the tests. For comparative purposes the absorption test affords a simple and sufficiently accurate means of obtaining the desired information. As applied to slate this test requires somewhat more care and precision than many other materials because of its dense nature and, consequently, the small quantities to be dealt with. Furthermore, the cleavage of slate—that property which permits it to be split into thin sheets of uniform thickness—must be taken into consideration when this test is made. On this account misleading results are often obtained on cubical specimens due to accidental cleavage cracks in the specimens. The shapes of specimens and larger number of specimens recommended in the following procedure are intended to eliminate to a large extent the inconsistent results which may be obtained on this material.

Scope

1. This method of test covers the procedure for determining the water absorption of slate.

Test Specimens

2. (*a*) The test specimens shall consist of square or rectangular slabs from $\frac{3}{16}$ to $\frac{5}{16}$ in. in thickness and not less than 4 in. on any side.

(*b*) Not less than six specimens shall be prepared from each sample of slate, the sample being considered as any number of pieces selected to represent a definite part or grade of the deposit.

Preparation of Specimens

3. (*a*) The slate shall be split to the required thickness and sawed to size. When the specimens are prepared from shingles no saw cut shall be nearer than 1 in. to the sheared edge of the shingle.

(*b*) The specimens shall be freed from loose particles by scrubbing with a fiber brush and clean water.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-18 on Natural Building Stones and Slate.

[2] Prior to adoption as standard, this method was published as tentative from 1925 to 1931, being revised in 1927 and 1931. Editorially revised and rearranged in 1939.

Procedure, Preferred Method

4. (*a*) The specimens shall be dried in a well ventilated oven at 110 ± 3 C. for 24 hr.

(*b*) After drying, the specimens may be cooled in the room for 15 min. and then weighed. When the specimens cannot be weighed immediately after cooling, they should be placed in a desiccator. The weight shall be determined to the nearest 0.01 g.

(*c*) The specimens shall be entirely immersed in distilled water at approximately 20 C. for 48 hr., then removed one at a time, the surface wiped off with a damp cloth, and each specimen immediately weighed to the nearest 0.01 g.

NOTE.—The operator should distinguish between a damp cloth and a wet one. In starting the operation, the towel should be sprinkled lightly. When the surface of the specimen is properly wiped off, it should appear dry.

Procedure, Alternative Method

5. Occasionally it is desirable to obtain results in a shorter period than that required by the procedure described in Section 4. In such cases, the 48-hr. immersion period may be supplanted by an 8-hr. boiling period (Note). In this alternative method the operations shall be the same as described in Section 4 up to that of immersion. Instead of allowing the specimens to soak for 48 hr. they may be placed in an enameled pan or other suitable vessel, covered with water, and boiled for 8 hr. Before the final weighing, the specimens shall be cooled by allowing tap water to flow over them for at least 30 min.

NOTE.—Boiling for 8 hr. has been found to give practically the same saturation as immersion for 48 hr.

Calculations and Report

6. (*a*) The percentage of absorption shall be calculated as follows:

$$\text{Absorption, per cent} = \frac{B - A}{A} \times 100$$

where:

A = weight of the dried specimen, and

B = weight of the specimen after immersion.

(*b*) In general, the average of all the tests shall be reported as the absorption of the slate. Individual determinations which differ by more than 25 per cent from the average of all the determinations may be disregarded when there is evidence that such results are of uncertain value. The absorption shall then be reported as the average of the remaining determinations, but all determinations shall be reported as information.

Standard Definition of

THE TERM SLATE[1]

A.S.T.M. Designation: C 119 - 27

ADOPTED, 1927.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 119; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Slate.—A microgranular crystalline stone derived from argillaceous sediments by regional metamorphism, and characterized by a perfect cleavage entirely independent of original bedding, which cleavage has been induced by pressure within the earth.

NOTE 1.—The essential mineral constituents are white mica (chiefly sericite) and quartz. Prominent accessory constituents are black mica (biotite), chlorite and hematite. Minor accessory constituents are carbonates, magnetite, apatite clay, andalusite, barite, rutile, pyrite, graphite, feldspar, zircon, tourmaline, and carbonaceous matter.

NOTE 2.—Igneous slates because of their rare occurrence and insignificant commercial importance are not covered in this definition.

[1] Under the standardization procedure of the Society, this definition is under the jurisdiction of the A.S.T.M. Committee C-18 on Natural Building Stones and Slate.

[2] Prior to adoption as standard, this definition was published as tentative from 1926 to 1927.

Standard Specifications for

AGGREGATE FOR MASONRY MORTAR[1]

A.S.T.M. Designation: C 144 - 44

ADOPTED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 144; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover aggregates for use in masonry mortar.

Material

2. Aggregate for use in masonry mortar shall consist of fine granular material composed of hard, strong, durable mineral particles which are free of injurious amounts of saline, alkaline, organic, or other deleterious substances.

Grading

3. (*a*) Aggregates for use in masonry mortar shall be graded from fine to coarse within the following limits, except as modified in accordance with Paragraphs (*b*) and (*c*):

Sieve Size	Percentages Passing Each Sieve
No. 4 (4760-micron)	100
No. 8 (2380-micron)	95 to 100
No. 16 (1190-micron)	60 to 100
No. 30 (590-micron)	35 to 70
No. 50 (297-micron)	15 to 35
No. 100 (149-micron)	0 to 15

(*b*) The fine aggregate shall be so graded that neither the proportion finer than a No. 16 (1190-micron) sieve and coarser than a No. 30 (590-micron) sieve nor the proportion finer than a No. 30 (590-micron) sieve and coarser than a No. 50 (297-micron) sieve exceeds 50 per cent.

(*c*) The requirements for grading in Paragraphs (*a*) and (*b*) represent the extreme limits which shall determine the suitability of a fine aggregate for use in masonry mortar. The gradation of material from any one source shall be reasonably uniform and shall not be permitted to vary over the extreme range shown in Paragraph (*a*). Fine aggregate from any one source having a variation in fineness modulus greater than plus or minus 0.20 from the fineness modulus of a representative sample submitted by the contractor shall be rejected, or it may be accepted subject to such adjustment in proportions as may be necessary by reason of changes in grading.

NOTE.—For heavy construction employing joints thicker than ½ in. a coarser aggregate may be desirable; for such work a fine aggregate

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M Committee C-12 on Mortars for Unit Masonry.

[2] Prior to adoption as standard, these specifications were published as tentative from 1939 to 1944, being revised in 1942.

conforming to conventional specifications for aggregate for use in concrete and containing not less than 10 per cent of material passing a No. 50 (297-micron) sieve is satisfactory. For unusually thin joints, such as occur for units having cut or ground edges, an aggregate conforming to the requirements of these specifications, but with not less than 95 per cent passing a No. 16 (1190-micron) sieve, should be used.

Deleterious Substances

4. (*a*) The amount of deleterious substances shall not exceed the following:

	Maximum Permissible Percentage by Weight
Coal and lignite	0.25
Clay lumps	1
Shale, alkali, coated grains, soft or flaky particles	1
Other deleterious substances	as specified
Total deleterious substances	3

(*b*) Aggregate subjected to the colorimetric test for organic impurities and producing a color darker than the standard shall be rejected unless it shall pass the mortar strength test as specified in Section 5.

Mortar Strength Test

5. Aggregate shall be of such quality when subjected to the mortar strength test that it shall have a compressive strength at ages of 7 and 28 days of not less than 95 per cent of that developed by a mortar of the same water-cement ratio and consistency made with the same cement and graded Ottawa sand having a fineness modulus of 2.40 ± 0.10.

Methods of Testing

6. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials:

(*a*) *Sieve Analysis.*—Standard Method of Test for Sieve Analysis of Fine and Coarse Aggregates (A.S.T.M. Designation: C 136).[3]

(*b*) *Washing Test.*—Standard Method of Test for Amount of Material Finer than No. 200 Sieve in Aggregates (A.S.T.M. Designation: C 117).[3]

(*c*) *Coal and Lignite.*—Standard Method of Test for Coal and Lignite in Sand (A.S.T.M. Designation: C 123).[3]

(*d*) *Clay Lumps.*—Standard Method of Test for Clay Lumps in Aggregates (A.S.T.M. Designation: C 142).[3]

(*e*) *Organic Impurities.*—Standard Method of Test for Organic Impurities in Sands for Concrete (A.S.T.M. Designation: C 40).[3]

(*f*) *Mortar Strength.*—Standard Method of Test for Structural Strength of Fine Aggregate Using Constant Water-Cement-Ratio Mortar (A.S.T.M. Designation: C 87).[3]

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: A2.1-1942

Standard Methods of
FIRE TESTS OF BUILDING CONSTRUCTION AND MATERIALS[1]

A.S.T.M. Designation: C 19 – 41

ADOPTED, 1933; REVISED 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 19; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

The performance of walls, columns, floors, and other building members under fire exposure conditions is an item of major importance in securing constructions that are safe, and that are not a menace to neighboring structures nor to the public. Recognition of this is registered in the codes of many authorities, municipal and other. It is important to secure balance of the many units in a single building, and of buildings of like character and use in a community; and also to promote uniformity in requirements of various authorities throughout the country. To do this it is necessary that the fire-resistive properties of materials and assemblies be measured and specified according to a common standard expressed in terms that are applicable alike to a wide variety of materials, situations, and conditions of exposure.

Such a standard is found in the methods that follow. They prescribe a standard exposing fire of controlled extent and severity. Performance is defined as the period of resistance to standard exposure elapsing before the first critical point in behavior is observed. Results are reported in units in which field exposures can be judged and expressed.

The methods may be cited as the "Standard Fire Tests," and the performance or exposure shall be expressed as "2-hr.," "6-hr.," "½-hr.," etc.

When a factor of safety exceeding that inherent in the test conditions is desired, a proportional increase should be made in the specified time-classification period.

Classifications of assemblies involving combustibles in such kind or quantity or so contained as to burn freely during the exposure to the test fire, or continue flaming or ignite after the furnace fire is shut off, shall be designated by the term "combustible" after the period assigned.

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[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee C-5 on Fire Tests of Materials and Construction.

These methods, of which the present standard represents a revision, were prepared by Sectional Committee A2 on Fire Tests of Materials and Construction, under the joint sponsorship of the National Bureau of Standards, the A.S.A. Fire Protection Group, and the American Society for Testing Materials, functioning under the procedure of the American Standards Association.

[2] Prior to their present adoption as standard, these methods were published as tentative from 1917 to 1918, being revised in 1918. They were adopted in 1918, but withdrawn and republished as tentative from 1926 to 1933, being revised in 1933.

Scope

1. (*a*) These methods of fire tests are applicable to assemblies of masonry units and to composite assemblies of structural materials for buildings, including bearing and other walls and partitions, columns, girders, beams, slabs, and composite slab and beam assemblies for floors and roofs. They are also applicable to other assemblies and structural units that constitute permanent integral parts of a finished building.

(*b*) It is the intent that classifications shall register performance during the period of exposure and shall not be construed as having determined suitability for use after fire exposure.

CONTROL OF FIRE TESTS

Time-Temperature Curve

2. The conduct of fire tests of materials and construction shall be controlled by the standard time-temperature curve shown in Fig. 1. The points on the curve that determine its character are:

1000 F. (538 C.)	at	5 min.
1300 F. (704 C.)	at	10 min.
1550 F. (843 C.)	at	30 min.
1700 F. (927 C.)	at	1 hr.
1850 F. (1010 C.)	at	2 hr.
2000 F. (1093 C.)	at	4 hr.
2300 F. (1260 C.)	at	8 hr. or over

Furnace Temperatures

3. (*a*) The temperature fixed by the curve shall be deemed to be the average temperature obtained from the readings of not less than five thermocouples symmetrically disposed and distributed to show the temperature near all parts of the sample, the thermocouples being enclosed in sealed porcelain tubes $\frac{3}{4}$ in. in outside diameter and $\frac{1}{8}$ in. in wall thickness, or, as an alternative in the case of base metal thermocouples, enclosed in sealed, standard-weight $\frac{1}{2}$-in. black wrought steel or black wrought iron pipe. The exposed length of the pyrometer tube and thermocouple in the furnace chamber shall be not less than 12 in. Other types of protecting tubes or pyrometers may be used that, under test conditions, give the same indications as the above standard within the limit of accuracy that applies for furnace-temperature measurements. For floors and columns, the junction of the thermocouples shall be placed 12 in. away from the exposed face of the sample at the beginning of the test and, during the test, shall not touch the sample as a result of its deflection. In the case of walls and partitions, the thermocouples shall be placed 6 in. away from the exposed face of the sample at the beginning of the test, and shall not touch the sample during the test, in the event of deflection.

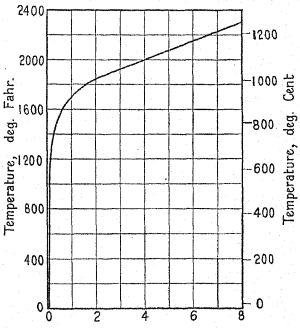


FIG. 1.—Time-Temperature Curve.

(*b*) The temperatures shall be read at intervals not exceeding 5 min. during the first hour, and thereafter the intervals may be increased to not more than 15 min.

(*c*) The accuracy of the furnace control shall be such that the area under the time-temperature curve, obtained by averaging the results from the pyrometer readings, is within 10 per cent of the corresponding area under the standard time-temperature curve shown in Fig. 1

for fire tests of 1 hr. or less duration, within 7.5 per cent for those over 1 hr. and not more than 2 hr., and within 5 per cent for tests exceeding 2 hr. in duration.

Temperatures of Unexposed Surfaces of Floors, Walls, and Partitions

4. (*a*) Temperatures at unexposed surfaces shall be measured with thermocouples or thermometers[3] placed under flexible, oven-dry, felted asbestos pads 6 in. square, 0.4 in. in thickness, and weighing not less than 1.0 nor more than 1.4 lb. per sq. ft. The pads shall be sufficiently soft so that, without breaking, they may be shaped to contact over the whole surface against which they are placed. The wire leads of the thermocouple or the stem of the thermometer shall have an immersion under the pad and be in contact with the unexposed surface for not less than $3\frac{1}{2}$ in. The hot junction of the thermocouple or the bulb of the thermometer shall be placed approximately under the center of the pad. The outside diameter of protecting or insulating tubes, and of thermometer stems, shall be not more than $\frac{5}{16}$ in. The pad shall be held firmly against the surface, and shall fit closely about the thermocouples or thermometer stems Thermometers shall be of the partial-immersion type, with a length of stem, between the end of the bulb and the immersion mark, of 3 in. The wires for the thermocouple in the length covered by the pad shall be not heavier than No. 18 B. & S. gage (0.04 in.) and shall be electrically insulated with heat-resistant and moisture-resistant coatings.

(*b*) Temperature readings shall be taken at not less than five points on the surface, one of which shall be approximately at the center, and four at approximately the centers of the quarter sections. If additional points are used they shall be symmetrically disposed about the center, with no location nearer than $1\frac{1}{2}$ times the thickness of the construction, or nearer than 12 in., to the edges. None shall be located opposite or on top of beams, girders, pilasters, or other structural members.

(*c*) Temperature readings shall be taken at intervals not exceeding 15 min. until a reading exceeding 212 F. (100 C.) has been obtained at any one point. Thereafter the readings may be taken more frequently at the discretion of the testing body, but the intervals need not be less than 5 min.

(*d*) Where the conditions of acceptance place a limitation on the rise of temperature of the unexposed surface, the temperature end point of the fire endurance period shall be determined by the average of the measurements taken at individual points; except that if a temperature rise 30 per cent in excess of the specified limit occurs at any one of these points, the remainder shall be ignored and the fire endurance period judged as ended.

CLASSIFICATION AS DETERMINED BY TEST

Report of Results

5. Results shall be reported in accordance with the performance in the tests prescribed in these specifications. They shall be expressed in time periods of resistance, as for example 4-hr., $\frac{1}{2}$-hr., etc.

TEST STRUCTURES

Test Structures

6. (*a*) The test structure may be located at any place where all the necessary facilities for properly conducting the test are provided.

(*b*) Entire freedom is left to each investigator in the design of the test structure and the nature and use of fuel, provided the test requirements are met.

[3] Under certain conditions it may be unsafe or impracticable to use thermometers.

TEST SAMPLE

Test Sample

7. (*a*) The test sample shall be truly representative of the construction for which classification is desired, as to materials, workmanship, and details such as dimensions of parts, and shall be built under conditions representative of those obtaining as practically applied in building construction and operation. The physical properties of the materials and ingredients used in the test sample shall be determined and recorded.

(*b*) The size and dimensions of the test sample specified herein are intended to apply for rating constructions of dimensions within the usual general range employed in buildings. If the conditions of use limit the construction to smaller dimensions, a proportionate reduction may be made in the dimensions of the samples for a test qualifying them for such restricted use.

CONDUCT OF FIRE TESTS

Fire Endurance Test

8. The fire endurance test on the sample with its applied load, if any, shall be continued until failure occurs, or until it has withstood the test conditions for a period equal to that herein specified in the conditions of acceptance for the given type of construction.

Hose Stream Test

9. (*a*) Where required by the conditions of acceptance, a duplicate sample shall be subjected to a fire exposure test for a period equal to one half of that indicated as the resistance period in the fire endurance test, but not for more than 1 hr., immediately after which the sample shall be subjected to the impact, erosion, and cooling effects of a hose stream directed first at the middle and then at all parts of the exposed face, changes in direction being made slowly.

(*b*) *Exemption.*—The hose stream test shall not be required in the case of indicated in the fire endurance test, of less than 1 hr.

(*c*) *Optional Program.*—The submitter may elect, with the advice and consent of the testing body, to have the hose stream test made on the sample subjected to the fire endurance test and immediately following the expiration of the fire endurance test.

(*d*) *Stream Equipment and Details.*—The stream shall be delivered through 2½-in. hose discharging through a National Standard Playpipe of corresponding size equipped with a 1⅛-in. discharge tip of the standard-taper smooth-bore pattern without shoulder at the orifice. The water pressure and duration of application shall be as prescribed in Table I.

TABLE I.

Resistance Period	Water Pressure at Base of Nozzle, psi.	Duration of Application, min. per 100 sq. ft. exposed area
8 hr. and over	45	6
4 hr. and over if less than 8 hr.	45	5
2 hr. and over if less than 4 hr.	30	2½
1½ hr. and over if less than 2 hr.	30	1½
1 hr. and over if less than 1½ hr.	30	1

(*e*) *Nozzle Distance.*—The nozzle orifice shall be 20 ft. from the center of the exposed surface of the test sample if the nozzle is so located that when directed at the center its axis is normal to the surface of the test sample. If otherwise located, its distance from the center shall be less than 20 ft. by an amount equal to 1 ft. for each 10 deg. of deviation from the normal.

Time of Testing

10. The material or construction shall not be tested until a large proportion of its final strength has been attained, and,

has been given off; this will usually require about 30 days time under favorable drying conditions. Artificial drying at temperatures not injurious to the material or construction to be tested may be used.

TESTS OF BEARING WALLS AND PARTITIONS

Size of Sample

11. The area exposed to fire shall be not less than 100 sq. ft., with neither dimension less than 9 ft. The test specimen shall not be restrained on its vertical edges.

Loading

12. During the fire endurance and fire and hose stream tests the construction shall be loaded in a manner calculated to develop theoretically, as nearly as practicable, the working stresses contemplated by the design.

Conditions of Acceptance

13. The test shall not be regarded as successful unless the following conditions are met:

(*a*) The wall or partition shall have sustained the applied load during the fire endurance test without passage of flame or gases hot enough to ignite cotton waste, for a period equal to that for which classification is desired.

(*b*) The wall or partition shall have sustained the applied load during the fire and hose stream test as specified in Section 9, without passage of flame, of gases hot enough to ignite cotton waste, or of the hose stream, and after cooling but within 72 hr. after its completion shall sustain a total load equal to the dead load plus twice the superimposed load specified above.

(*c*) Transmission of heat through the wall or partition during the fire endurance test shall not have been such as to raise the temperature on its unexposed surface more than 250 F. (139 C.) above its initial temperature.

TESTS OF NONBEARING WALLS AND PARTITIONS

Size of Sample

14. The area exposed to fire shall be not less than 100 sq. ft., with neither dimension less than 9 ft. The test specimen shall be restrained on all four edges.

Conditions of Acceptance

15. The test shall not be regarded as successful unless the following conditions are met:

(*a*) The wall or partition shall have withstood the fire endurance test without passage of flame or gases hot enough to ignite cotton waste, for a period equal to that for which classification is desired.

(*b*) The wall or partition shall have withstood the fire and hose stream test as specified in Section 9, without passage of flame, of gases hot enough to ignite cotton waste, or of the hose stream.

(*c*) Transmission of heat through the wall or partition during the fire endurance test shall not have been such as to raise the temperature on its unexposed surface more than 250 F. (139 C.) above its initial temperature.

TESTS OF COLUMNS

Size of Sample

16. The length of the column exposed to fire shall, when practicable, approximate the maximum clear length contemplated by the design, and for building columns shall be not less than 9 ft. The contemplated details of connections, and their protection if any, shall be applied according to the methods of acceptable field practice.

Loading

17. (*a*) During the fire endurance test the column shall be exposed to fire on all sides and shall be loaded in a manner calculated to develop theoretically, as nearly as practicable, the working stresses contemplated by the design. Provision shall be made for transmitting the load to the exposed portion of the column without unduly increasing the effective column length.

(*b*) If the submitter and the testing body jointly so decide, the column may be subjected to $1\frac{3}{4}$ times its designed working load before the fire endurance test is undertaken. The fact that such a test has been made shall not be construed as having had a deleterious effect on the fire endurance test performance.

Condition of Acceptance

18. The test shall not be regarded as successful unless the column shall have sustained the applied load during the fire endurance test for a period equal to that for which classification is desired.

TESTS OF FLOORS AND ROOFS

(The following contemplates application of fire exposure to the under side of constructions.)

Size of Sample

19. The area exposed to fire shall be not less than 180 sq. ft., with neither dimension less than 12 ft. Beams or girders, if a part of the construction under test, shall lie within the combustion chamber and have a clearance of not less than 8 in. from its walls.

Loading

20. During the fire endurance and fire and hose stream tests the construction shall be loaded in a manner calculated to develop theoretically, as nearly as practicable, the working stresses in each member contemplated by the design.

Conditions of Acceptance

21. The test shall not be regarded as successful unless the following conditions are met:

(*a*) The construction shall have sustained the applied load during the fire endurance test without passage of flame or gases hot enough to ignite cotton waste, for a period equal to that for which classification is desired.

(*b*) The construction shall have sustained the applied load during the fire and hose stream test as specified in Section 9, without passage of flame, of gases hot enough to ignite cotton waste, or of the hose stream, and, after cooling but within 72 hr. after its completion, shall sustain a total load equal to the dead load plus twice the superimposed load specified above.

(*c*) Transmission of heat through the construction during the fire endurance test shall not have been such as to raise the temperature on its unexposed surface more than 250 F. (139 C.) above its initial temperature.

TESTS OF PROTECTION FOR COMBUSTIBLE FRAMING, OR FOR COMBUSTIBLE FACINGS ON THE UNEXPOSED SIDE OF WALLS, PARTITIONS, AND FLOORS

Character of Sample

22. Test panels carrying wall, partition, or floor protection shall be finished with the protections which are the subject of the test, except that where the finish on the unexposed side is not the subject of the test and is not specifically indicated, the testing authority shall apply a finish judged suitable for the purpose. In case a floor construction, as installed for actual use, is to have no finish on the unexposed side, it shall be so tested.

Size of Sample

23. The area exposed to fire shall be, for tests of wall and partition protection, not less than 100 sq. ft. with neither dimension less than 9 ft.; for tests of floor protection, not less than 180 sq. ft. with neither dimension less than 12 ft.

Conditions of Acceptance

24. The test shall not be regarded as successful unless the following conditions are met:

(*a*) The protection shall have withstood the fire endurance test, without ignition of the materials protected, for a period equal to that for which classification is desired.

(*b*) Transmission of heat through the protection during the fire endurance test shall not have been such as to raise the temperatures at its contact with the protected structural members or facings of the test panel more than 250 F. (139 C.) above the initial temperatures at these points, except that for members closely embedded on three sides in masonry, concrete, or other incombustible materials the permissible temperature rise may be 325 F. (181 C.).

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: A2.2-1942

Standard Methods of
FIRE TESTS OF DOOR ASSEMBLIES[1]

A.S.T.M. Designation: C 152 – 41

ADOPTED, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 152; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. (*a*) These methods of fire test are applicable to door assemblies, of various materials and of various types of construction, for installation as wall opening protectives to serve against the passage of fire, heat, fumes, or smoke.[3]

(*b*) It is the intent that classifications shall register performance during the period of exposure and shall not be construed as having determined suitability for use after exposure to either fire, or fire and hose stream.

(*c*) These methods do not establish ratings, or standards of performance for different uses, as these depend upon service requirements and are under the control of various regulatory bodies.

CONTROL OF FIRE TESTS

Time-Temperature Curve

2. The conduct of fire tests of materials and construction shall be controlled by the standard time-temperature curve shown in Fig. 1. The points on the curve that determine its character are:

1000 F. (538 C.)	at 5 min.
1300 F. (704 C.)	at 10 min.
1550 F. (843 C.)	at 30 min.
1700 F. (927 C.)	at 1 hr.
1850 F. (1010 C.)	at 2 hr.
2000 F. (1093 C.)	at 4 hr.
2300 F. (1260 C.)	at 8 hr. or over

Furnace Temperatures

3. (*a*) The temperature fixed by the curve shall be deemed to be the average temperature obtained from the readings of not less than five thermocouples symmetrically disposed and distributed to show the temperature near all parts of the test assembly, the thermocouples being enclosed in sealed porcelain tubes $\frac{3}{4}$ in. in outside diameter and $\frac{1}{8}$ in. in wall thickness, or, in the case of base metal thermocouples, enclosed in sealed, standard-weight $\frac{1}{2}$-in. wrought steel pipe. The exposed length of the pyrometer tube and couple in the furnace chamber shall be not less than 12 in. Other types of protecting tubes or pyrometers may be used that, under test conditions, give the same indications as the above standard within the limit of accuracy that applies for furnace-temperature

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee C-5 on Fire Tests of Materials and Construction.

[2] Prior to adoption as standard, these methods were published as tentative from 1940 to 1941.

[3] The committee is giving consideration to the subject of smoke generation and the creation and spread of dangerous fumes, which upon completion will result in subsequent revision of these methods.

measurements. The junction of the thermocouples shall be placed 4 to 6 in. away from the exposed face of the test assembly at the beginning of the test, and during the test shall not touch the assembly as a result of its deflection.

(*b*) The temperatures shall be read at intervals not exceeding 5 min. during the first hour, and thereafter at intervals not exceeding 15 min.

(*c*) The accuracy of the furnace control shall be such that the area under the time-temperature curve, obtained by averaging the results from the

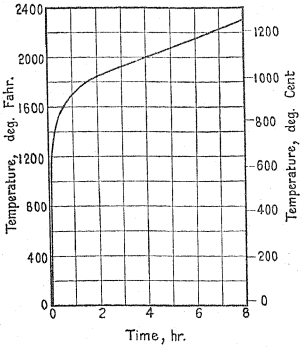


FIG. 1.—Time-Temperature Curve.

pyrometer readings, is within 10 per cent of the corresponding area under the standard time-temperature curve shown in Fig. 1 for fire tests of 1 hr. or less duration, within 7.5 per cent for those over 1 hr. and not more than 2 hr., and within 5 per cent for tests exceeding 2 hr. in duration.

Temperature of Unexposed Surfaces of Doors

4. (*a*) Temperatures at unexposed surfaces shall be measured with thermocouples or thermometers placed under flexible, oven-dry, felted asbestos pads 6 in. square, 0.4 in. in thickness, and weighing not less than 1.0 nor more than 1.4 lb. per sq. ft. The pads shall be sufficiently soft that, without breaking, they may be shaped to contact over the whole surface against which they are placed. The wire leads of the thermocouple or the stem of the thermometer shall have an immersion under the pad and be in contact with the unexposed surface for not less than $3\frac{1}{2}$ in. The hot junction of the thermocouple or the bulb of the thermometer shall be placed approximately under the center of the pad. The outside diameter of protecting or insulating tubes, and of thermometer stems, shall be not more than $\frac{5}{16}$ in. The pad shall be held firmly against the surface, and shall fit closely about the thermocouples or thermometer stems. Thermometers shall be of the partial-immersion type, with a length of stem, between the end of the bulb and the immersion mark, of 3 in. The wires of the thermocouple in the length covered by the pad shall be not heavier than No. 18 B. & S. gage (0.04 in.) and shall be electrically insulated with heat-resistant and moisture-resistant coatings.

(*b*) Temperature readings shall be taken at not less than three points, one located at the middle of the unexposed surface of the test assembly and one each at the mid-points of the upper and lower halves of the assembly. If the construction is not uniform, or if, in the opinion of the testing authority, temperature measurements at other locations are necessary to give a true indication of performance, the thermocouples shall be symmetrically disposed to give results representative of the different constructions, and preferably with no location nearer than 12 in. to the edges of the test assembly.

(*c*) Temperature readings on the unexposed surfaces shall be taken at the same intervals as specified for furnace temperatures.

(*d*) If a temperature end point of the rating period is specified, it shall be determined by the average of the measurements taken at individual points representing the least resistant construction; except that if a temperature rise 30 per cent in excess of the limit established by the regulatory body for whom the test is made occurs at any one of these points, the remainder shall be ignored and the fire endurance period judged as ended.

NOTE.—At the discretion of the testing authority, additional temperature measurements may be taken on, or near the unexposed surface.

TEST STRUCTURES

Test Structures

5. (*a*) The test structure may be located at any place where all the necessary facilities for properly conducting the test are provided.

(*b*) Entire freedom is left to each testing authority in the design and location of the test structure and the nature and use of fuel, provided the test requirements are met.

TEST ASSEMBLIES

Test Assemblies

6. (*a*) The test assembly shall be truly representative as to material, details (installation, hardware trim, and finish), and workmanship of the construction for which classification is desired. A record of materials and construction details, adequate for identification, shall be made.

(*b*) The test assembly shall consist of the door or doors, together with the necessary framing and appurtenances, installed in a single opening on one or both sides of a common wall.

Size of Test Assembly

7. The classification or rating derived from the test assembly shall be considered to apply to doors of similar design and construction, smaller in dimension and in area, or whose area does not exceed that of the test assembly by more than 25 per cent.

Mounting of Test Assembly

8. (*a*) Swinging doors shall be mounted so as to open into the furnace chamber. Sliding and rolling doors, unless of the flush-mounted type, shall be mounted on the fire side of the wall. Clearances of swinging doors of hollow metal or kalamein types shall conform to good practice,[4] but shall not be less than $\frac{3}{32}$ in. between the door and hinge and lock jambs, $\frac{1}{16}$ in. at the top, and $\frac{3}{16}$ in. at the bottom.

(*b*) Minimum clearances for swinging wood doors shall be $\frac{1}{16}$ in. at the sides and top, and $\frac{3}{16}$ in. at the bottom.

(*c*) If special tightness against the passage of smoke is desired, or if other clearances are used for any reason, the actual clearances shall be reported in the classification of the test assembly.

(*d*) Sliding and rolling doors of any type shall fit well against wall surfaces or in guides, but retaining methods shall allow free and easy movement of the door from any position.

CONDUCT OF FIRE TESTS

Fire Test

9. (*a*) During the fire test the pressure within the furnace shall be maintained as nearly equal to the outside atmospheric pressure as possible.

(*b*) The fire test on the test assembly shall be continued until failure occurs, or the limit of resistance specified by the submitter has been met.

[4] Hollow Metal Single-Acting Swing Doors, Frames, and Trim, Dept. of Commerce Simplified Practice Recommendation R82-28, and Kalamein Single-Acting Swing Doors, Frames, and Trim, Dept. of Commerce Simplified Practice Recommendation R83-28.

Hose Stream Test

10. (*a*) Unless the optional program of the following Paragraph (*c*) is specified, a duplicate test assembly shall be subjected to a fire exposure test for a period equal to one half of the fire resistance period in the fire endurance test, but not for more than 1 hr., immediately after which the test assembly shall be subjected to the impact, erosion, and cooling effects of a hose stream directed first at the middle and then at all parts of the exposed face, changes in direction being made slowly.

(*b*) *Exemption.*—The hose stream test shall not be required in the case of the opening protective having a resistance period, indicated in the fire resistance test, of less than 1 hr.

(*c*) *Optional Program.*—When the test assembly is tested for a desired time limit of fire resistance, the submitter may elect, with the advice and consent of the testing authority, to have the hose stream test made on the assembly subjected to the fire resistance test and immediately following the expiration of the fire resistance test.

(*d*) *Stream Equipment and Details.*—The stream shall be delivered through $2\frac{1}{2}$-in. hose discharging through a National Standard Playpipe of corresponding size equipped with a $1\frac{1}{8}$-in. discharge tip of the standard-taper smooth-bore pattern without shoulder at the orifice. The water pressure and duration of application shall be as prescribed in Table I.

TABLE I.

Resistance Period	Water Pressure at Base of Nozzle, psi.	Duration of Application, min. per 100 sq. ft. exposed area[a]
4 hr. and over	45	5
2 hr. and over, if less than 4 hr.	30	$2\frac{1}{2}$
1 hr. and over, if less than 2 hr.	30	$1\frac{1}{2}$
Less than 1 hr. (if desired)	30	1

[a] A 1-min. application of the hose stream shall be the minimum.

(*e*) *Nozzle Distance.*—The nozzle orifice shall be 20 ft. from the center of the exposed surface of the test assembly if the nozzle is so located that when directed at the center its axis is normal to the surface of the test assembly. If otherwise located, its distance from the center shall be less than 20 ft. by an amount equal to 1 ft. for each 10 deg. of deviation from the normal.

Time of Testing

11. Masonry settings shall be allowed to dry for at least 3 days before tests are made.

CONDITIONS OF ACCEPTANCE

Heat Insulation

12. The testing authority shall record and include in the test report, the temperature measurements taken within the furnace and on the unexposed side of the test assembly as specified in Sections 3 (*a*) and (*b*), and 4 (*a*) to (*d*). Observations of features of the test having a bearing upon the performance of the test assembly shall also be made a part of the report, including passage or production of fumes and smoke.

Fire Resistance

13. The fire resistance test shall not be regarded as successful unless the following conditions are met:

(*a*) The test assembly shall have remained securely in the opening during the fire exposure period and, if required, during the hose stream test. When the test assembly comprises two doors located one on each face of a common wall, the test conditions are met when the door on the unexposed side remains securely in the opening, irrespective of the condition of the door on the fire side.

(*b*) The test assembly shall have withstood the fire endurance and hose stream test, except that small portions

of glass dislodged by the hose stream shall not be considered a weakness, without developing openings anywhere through the assembly, or openings markedly in excess of the initial clearances at the outside or meeting edges.

(*c*) If a transmitted temperature end point is specified, it shall be considered as a condition of acceptance.

Standard Methods of

SAMPLING AND MIXING THERMAL INSULATING CEMENT[1]

A.S.T.M. Designation: C 163 – 44

ADOPTED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 163; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods cover the procedures for sampling thermal insulating cement and for mixing the cement for preparation of specimens for use in all tests on the cement.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Mixing Surface or Pan.*—A nonabsorbent and corrosion-resistant surface or shallow pan approximately 3 ft. square.

(*b*) *Trowel.*—A 16-in. rectangular plasterer's trowel.

(*c*) *Scales.*—A pair of scales accurate to within 0.5 oz.

(*d*) *Water Container.*—A container suitable for holding approximately 2 gal. of water.

Mixing Water

3. The mixing water shall be equal in quality to that used for domestic purposes.

Sampling

4. At least 3 per cent of the packages comprising any one shipment shall be tested. They shall be systematically selected so as to be representative of all parts of the shipment. Each package shall be sampled by removing approximately $\frac{1}{4}$ lb. from the top and $\frac{1}{4}$ lb. from the center. The composite of all samples thus selected shall then be thoroughly mixed and quartered until the sample is of the desired size.

Procedure for Mixing

5. (*a*) The sample of dry cement shall be weighed and placed upon a smooth, nonabsorbent and corrosion-resistant surface.

(*b*) A crater shall be formed in the center of the dry cement and the mixing water shall be placed therein. The amount of mixing water used shall conform to the exact proportions recommended by the manufacturer of the cement.

(*c*) The material on the outer edge shall be turned into the crater by means of the plasterer's trowel.

(*d*) After the water has been ab-

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee C-16 on Thermal Insulating Materials.

[2] Prior to adoption as standard, these methods were published as tentative from 1941 to 1944.

sorbed by the cement or has remained in contact therewith for the period of time recommended by the manufacturer, the trowel shall be rapidly drawn over the cement by alternately applying and releasing pressure on its trailing edge.

(*e*) After three such passes of the trowel across the cement, it shall be gathered in at the edges and turned over with the trowel.

(*f*) The cycle described in Paragraphs (*d*) and (*e*) shall be repeated either until uniformity and optimum plasticity have been obtained or as specified by the manufacturer.

(*g*) The cement shall be molded immediately after mixing or after the lapse of such period of time as may be recommended by the manufacturer.

Standard Method of Test for

BULK DENSITY OF THERMAL INSULATING CEMENT[1]

A.S.T.M. Designation: C 164 - 44

ADOPTED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 164; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method covers the procedure for determining the bulk density of thermal insulating cement to permit the calculation of the necessary storage space for a given quantity of cement in packages as received.

Definition

2. The bulk density of an insulating cement is the package volume in cubic feet for 100 lb. of packaged cement.

Apparatus

3. The apparatus shall consist of the following:

(*a*) *Scales.*—A pair of scales accurate to within 1 oz.

(*b*) *Rule or Tape.*—An ordinary folding 6-ft. rule or a steel tape accurate to within plus or minus $\frac{1}{16}$ in.

(*c*) *Square.*—A carpenter's square having the blade (the broader, longer leg) extended to a length of at least 36 in.

Procedure

4. (*a*) Twelve packages shall be systematically selected so as to be representative of all parts of the shipment or stock to be tested. The packages may be weighed separately or in groups or they may be all weighed at one time, depending upon the capacity of the available scales. The twelve packages shall then be stacked horizontally in a corner against two walls in a single row three packages high. If the cement is packed in sacks, the tied mouths of the sacks shall be placed alternately against the wall and away from it. At least three measurements of the width, three of the height, and two of the length of the stack shall be made in accordance with the procedure described in the following Paragraphs (*b*) to (*d*). All dimensions shall be expressed in feet.

(*b*) *Width.*—At each of three positions equally spaced along the length of the stack, the tongue (the shorter, narrower arm) of the modified carpenter's square shall be placed on the floor perpendicular to the face of the stack with the extended blade perpendicular to the floor and touching the face of the stack. The shortest distance from the inner edge of the blade to the wall behind the stack shall be measured with the rule or tape. The average of these three measurements shall be taken as the width of the stack.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-16 on Thermal Insulating Materials.

[2] Prior to adoption as standard, this method was published as tentative from 1941 to 1944.

(*c*) *Height.*—At each of three positions equally spaced along the length of the stack, the tongue of the modified carpenter's square shall be placed against the wall above the top of the stack and perpendicular to the floor with the extended blade perpendicular to the wall and touching the top of the stack. The vertical distance from the lower edge of the blade to the floor shall be measured. The average of these three measurements shall be taken as the height of the stack.

(*d*) *Length.*—At each of two positions one fourth of the width of the stack from the front and rear faces of the stack, the tongue of the modified carpenter's square shall be placed on the floor parallel to the nearest wall against which the stack is placed and with the extended blade perpendicular to the floor and touching the end of the stack. The shortest distance from the inner edge of the blade to the wall at the opposite end of the stack shall be measured. The average of these two measurements shall be taken as the length of the stack.

Calculation

5. The bulk density in cubic feet per 100 lb. shall be calculated as follows:

$$\text{Bulk density} = \frac{w \times h \times l}{M} \times 100$$

where:

w = average width of stack in feet,
h = average height of stack in feet,
l = average length of stack in feet, and
M = gross weight of stack in pounds.

Standard Methods of Test for

THICKNESS AND DENSITY OF BLANKET TYPE THERMAL INSULATING MATERIALS[1]

A.S.T.M. Designation: C 167 – 44

ADOPTED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 167; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods of test cover the procedures for determining the thickness and density of flexible, felted or woven thermal insulating blankets, rolls, or batts, with or without reinforcement, composed of fibrous materials.

Apparatus

2. The apparatus for measuring thickness of the test specimen shall be a depth gage of the type shown in Fig. 1.

Test Specimen

3. (*a*) The size of the test specimen shall not be limited, but the specimen shall be of sufficient area to be representative of the lot or shipment in question.

(*b*) In case of reinforced blanket insulations, the reinforcing material, regardless of its nature, shall not be removed when making thickness measurements. However, in case of high-rib lath reinforcement, the thickness measurement shall be made between ribs or, where this is not practical, the thickness of the rib lath shall be deducted from the total thickness as measured with the depth gage.

Procedure

4. (*a*) *Thickness Measurements.*—The test specimen shall be ruled off into ten approximately square and equal areas and a thickness measurement taken at the center of each area. In making the thickness measurements the test specimen shall be placed on a flat surface and the penetrating pin of the depth gage shall be forced downward through the specimen, perpendicular to the flat surface. When the point of the pin touches the flat surface, the sliding disk shall be lowered to the point of contact with the top surface of the specimen. The gage shall be withdrawn and the distance from the point of the pin to the sliding disk measured within an accuracy of plus or minus $\frac{1}{16}$ in.

(*b*) *Weight and Dimensional Measurements.*—The entire test specimen used for the thickness measurements shall be weighed within an accuracy of plus or minus 0.5 per cent, and the length and width of the specimen shall be measured within an accuracy of plus or minus 0.5 per cent.

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee C-16 on Thermal Insulating Materials.

[2] Prior to adoption as standard, these methods were published as tentative from 1941 to 1944.

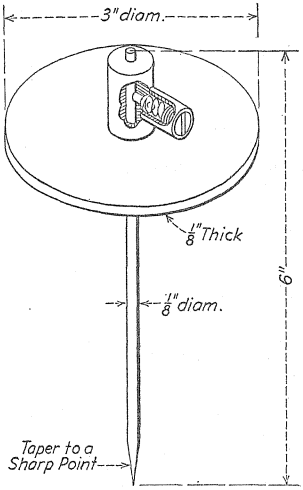


FIG. 1.—Depth Gage for Thickness Measurements.

Calculations

5. (*a*) *Thickness.*—The average of the ten thickness measurements made in accordance with Section 4 (*a*) shall be taken as the "thickness, as-received" of the test specimen.

(*b*) *Density.*—The "density, as-received" and the "density, at specified thickness" shall be calculated as follows:

$$\text{"Density, as received," lb. per cu. ft.} = \frac{W \times 1728}{A \times B \times C_1}$$

$$\text{"Density, at specified thickness," lb. per cu. ft.} = \frac{W \times 1728}{A \times B \times C_2}$$

where:

W = weight of test specimen in pounds,
A = length of specimen in inches,
B = width of specimen in inches,
C_1 = "thickness, as-received," and
C_2 = thickness specified on ordering or designating the material.

Report

6. The report shall include the following:

(*1*) Average, maximum, and minimum values of "thickness, as-received," expressed in inches.

(*2*) "Density, as-received," expressed in pounds per cubic foot.

(*3*) "Density, at specified thickness" expressed in pounds per cubic foot.

Standard Definitions of

TERMS RELATING TO THERMAL INSULATING MATERIALS[1]

A.S.T.M. Designation: C 168 – 44

ADOPTED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 168; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Preformed Thermal Insulating Block.—A rigid or semirigid thermal insulating material, either flat or segmental, for application as received, and excluding brick of the 9-in. series.

Thermal Insulating Cement.—A prepared material which, when mixed with a suitable proportion of water, applied as a plastic mass, and dried in place, affords a substantial resistance to heat transmission.

[1] Under the standardization procedure of the Society, these definitions are under the jurisdiction of the A.S.T.M. Committee C-16 on Thermal Insulating Materials.

[2] Prior to adoption as standard, these definitions were published as tentative from 1941 to 1944.

Standard Specifications for

REFRACTORIES FOR HEAVY DUTY STATIONARY BOILER SERVICE[1]

A.S.T.M. Designation: C 64 - 41

ADOPTED, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 64; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. (*a*) The purpose of these specifications is the selection of fireclay refractories for heavy duty boiler furnaces in which the temperature of the combustion chamber is continuously, or a significant portion of the time, above 2400 F. (1315 C.). They may not apply to special conditions sometimes encountered in high-power utility boilers.

(*b*) These specifications are intended to cover only burned products made from fire clay by the usual processes of manufacture, and are not intended to include products other than those regularly sold as fireclay brick.

Zones of Highest Temperatures

2. (*a*) The refractories to be used in the zones of highest temperatures, such as sidewalls, arches, doors, and jambs, shall be high heat duty or super duty fireclay brick. The conditions to be encountered in the particular installation should be carefully considered so as properly to select from the following classification (Paragraphs (*b*) to (*e*)) the type or types of refractories best suited for the service. Unless type *B*, *C*, or *D* is definitely specified, there shall be supplied for this section of the specifications, type *A*, which is designed for average boiler furnace conditions.

(*b*) *Average Boiler Furnace Conditions.*—In the case of average heavy duty boiler furnace conditions, the refractories shall conform to the requirements for type *A* (Section 6(*a*)).

(*c*) *Moderately Severe Conditions of Spalling.*—When moderately severe conditions of spalling are to be encountered, the refractories shall conform to the requirements for type *B* (Section 6(*b*)).

(*d*) *Moderately Severe Conditions of Slagging.*—When moderately severe con-

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-8 on Refractories.

[2] These specifications together with the Standard Specifications for Refractories for Moderate Duty Stationary Boiler Service (C 153 - 41) replace the former Standard Specifications for Fireclay Brick for Stationary Boiler Service (C 64 - 39). These specifications comprise a revision of the requirements for heavy duty refractories formerly contained in Standard Specifications C 64 - 39.

Prior to their present adoption as standard, these specifications were published as tentative from 1927 to 1928. They were adopted in 1928, published as standard from 1928 to 1934, but withdrawn in 1934 and republished as tentative from 1934 to 1936. They were again adopted in 1936, published as standard from 1936 to 1940, being revised in 1939, and again withdrawn and republished as tentative from 1940 to 1941. Editorially revised in 1942.

ditions of slagging are to be encountered, and provided the spalling condition is not too severe, the refractories shall conform to the requirements for type *C* (Section 6 (*c*)).

(*e*) *Extremely Severe Conditions of Spalling or Slagging.*—When extremely severe conditions of spalling or slagging are to be encountered, and especially when both may occur to an unusual extent, the use of super duty fireclay refractories may be justified. When specified, they shall conform to the requirements for type *D* (Section 6 (*d*)).

Division Walls Heated on Both Sides

3. Depending upon the particular conditions to be encountered, the refractories used in constructing the division walls shall be high heat duty or super duty fireclay brick. Unless the super duty fireclay brick, type *D* (Section 6 (*d*)) is definitely specified, there shall be supplied for this section of the specifications the high heat duty fireclay brick, type *E* (Section 6 (*e*)).

Zones of Lower Temperatures

4. This section of the specifications pertains to refractories to be used in backing up the brick in the zones of highest temperatures and for walls of the second and third passes. The service conditions to which such brick are subjected vary with the size and type of boiler, thickness of wall, degree of insulation, draft conditions, and the actual position in the furnace. Leaky baffles may sometimes cause the brick beyond the first tube bank to be subjected to higher temperatures than would normally be present. Depending upon the particular conditions, and upon the margin of safety desired, the brick selected shall be high, intermediate, or low heat duty fireclay brick as specified by type *A*, *F*, or *G*, respectively. Unless type *A* or *G* is definitely specified for this section of the specifications, there shall be supplied the intermediate heat duty brick, type *F* (Section 6 (*f*)).

REQUIREMENTS FOR THE VARIOUS TYPES OF REFRACTORIES

Size and Warpage

5. The following permissible variations in size and warpage shall apply to each type of refractory listed in Section 6:

(*a*) *Size.*—When tested in accordance with the Methods of Test for Size and Bulk Density of Refractory Brick (A.S.T.M. Designation: C 134) of the American Society for Testing Materials,[3] the standard 9-in. fireclay brick shall not show a size variation of more than plus or minus 2 per cent. Tile or shapes shall not show a size variation of more than plus or minus 2 per cent on dimensions of 4 in. or over, and plus or minus 3 per cent on dimensions smaller than 4 in.

(*b*) *Warpage.*—Ninety-five per cent of the shapes shall not show a warpage greater than 1 per cent of the diagonal used in making the measurement.

Test Requirements

6. (*a*) ***Type A.***—Type *A* shall be a high heat duty[4] fireclay brick conforming to the following requirements:

Refractoriness.—The brick shall be required to meet only *one* of the following requirements: Pyrometric cone equivalent not lower than cone No. 31–32, *or* not more than 1.5 per cent deformation in the 2460 F. (1350 C.) load test.

Permanent Linear Shrinkage.—When tested in accordance with schedule B (2550 F., 1400 C.) of the permanent linear change test, the brick shall not

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

[4] See the Classification of Fireclay Refractories (A.S.T.M. Designation: C 27), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

show a contraction of more than 1.5 per cent.

Spalling.—When subjected to the panel spalling test (preheated at 2910 F., 1600 C.), the brick shall not show a loss greater than 15 per cent.

(*b*) ***Type B.***—Type *B* shall be a high heat duty[4] fireclay brick conforming to the following requirements:

Refractoriness.—The brick shall be required to meet only *one* of the following requirements: Pyrometric cone equivalent not lower than cone No. 31-32, *or* not more than 1.5 per cent deformation in the 2460 F. (1350 C.) load test.

Permanent Linear Shrinkage.—When tested in accordance with schedule B (2550 F., 1400 C.) of the permanent linear change test, the brick shall not show a contraction of more than 1.5 per cent.

Spalling.—When subjected to the panel spalling test (preheated at 2910 F., 1600 C.), the brick shall not show a loss greater than 10 per cent.

(*c*) ***Type C.***—Type *C* shall be a high heat duty[4] fireclay brick conforming to the following requirements:

Refractoriness.—The brick shall be required to meet only *one* of the following requirements: Pyrometric cone equivalent not lower than cone No. 31-32, *or* not more than 1.5 per cent deformation in the 2460 F. (1350 C.) load test.

Permanent Linear Shrinkage.—When tested in accordance with schedule B (2550 F., 1400 C.) of the permanent linear change test, the brick shall not show a contraction of more than 1.5 per cent.

Water Absorption.—Because *two* varieties of clay refractories can be supplied to conform to the requirements for refractoriness as given above, each type shall conform to a specified maximum water absorption. The brick intended to conform to the specified P.C.E. shall have a water absorption of not more than 10 per cent. Brick which are supplied to conform to the load test requirement shall have a water absorption of not more than 18 per cent.

(*d*) ***Type D.***—Type *D* shall be a super duty[4] fireclay brick conforming to the following requirements:

Pyrometric Cone Equivalent.—The brick shall have a P.C.E. not lower than cone No. 33.

Permanent Linear Shrinkage.—When tested in accordance with schedule C (2910 F., 1600 C.) of the permanent linear change test, the brick shall not show a contraction of more than 1 per cent.

Spalling.—When subjected to the panel spalling test (preheated at 3000 F., 1650 C.), the brick shall not show a loss greater than 4 per cent.

(*e*) ***Type E.***—Type *E* shall be a high heat duty[4] fireclay brick having the following characteristics:

Refractoriness.—*Two* varieties of clay refractories are available for this class: Brick which shall show a deformation of not more than 6 per cent in the 2460 F. (1350 C.) load test and shall have a pyrometric cone equivalent not lower than cone No. 31-32; *or* brick which shall show a deformation of not more than 1.5 per cent in the 2460 F. (1350 C.) load test.

(*f*) ***Type F.***—Type *F* shall be an intermediate heat duty[4] fireclay brick conforming to the following requirements:

Refractoriness.—The brick shall be required to meet only *one* of the following requirements: Pyrometric cone equivalent not lower than cone No. 29, *or* not more than 3 per cent deformation in the 2460 F. (1350 C.) load test.

Modulus of Rupture.—The brick shall have a modulus of rupture of not less than 500 psi.

(*g*) ***Type G.***—Type *G* shall be a low heat duty[4] fireclay brick conforming to the following requirements:

Pyrometric Cone Equivalent.—The brick shall have a P.C.E. not lower than cone No. 19.

Modulus of Rupture.—The brick shall have a modulus of rupture of not less than 500 psi.

Retests

7. Because of variables resulting from sampling and the lack of perfect reproducibility in tests conducted by different laboratories, resampling and retesting may be carried out when requested by either the purchaser or the manufacturer. This may apply in instances where the first test result does not conform to the limits prescribed in these specifications. The final result to be used for each test shall be the average of the original and the retest figures.

NOTE.—Retest provisions for variation in size and water absorption are provided for in those test methods.

Methods of Testing

8. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials:

(*a*) *Size.*—Methods of Test for Size and Bulk Density of Refractory Brick (A.S.T.M. Designation: C 134).[3]

(*b*) *Warpage.*—Method of Test for Warpage of Refractory Brick and Tile (A.S.T.M. Designation: C 154).[3]

(*c*) *Pyrometric Cone Equivalent.*—Method of Test for Pyrometric Cone Equivalent (P.C.E.) of Refractory Materials (A.S.T.M. Designation: C 24).[3]

(*d*) *Load Test* (2460 F., 1350 C.).—Schedule No. 2 of the Method of Testing Fireclay Refractories Under Load at High Temperatures (A.S.T.M. Designation: C 16).[3]

(*e*) *Permanent Linear Shrinkage for High Heat Duty Brick* (2550 F., 1400 C.).—Schedule B of the Method of Test for Permanent Linear Change After Reheating of Refractory Brick (A.S.T.M. Designation: C 113).[3]

(*f*) *Permanent Linear Shrinkage for Super Duty Brick* (2910 F., 1600 C.).—Schedule C of the Method of Test for Permanent Linear Change After Reheating of Refractory Brick (A.S.T.M. Designation: C 113).[3]

(*g*) *Panel Spalling Test for High Heat Duty Brick* (2910 F., 1600 C.).—Method of Panel Test for Resistance to Thermal and Structural Spalling of High Heat Duty Fireclay Brick (A.S.T.M. Designation: C 107).[3]

(*h*) *Panel Spalling Test for Super Duty Brick* (3000 F., 1650 C.).—Method of Panel Test for Resistance to Thermal and Structural Spalling of Super Duty Fireclay Brick (A.S.T.M. Designation: C 122).[3]

(*i*) *Water Absorption.*—Methods of Test for Apparent Porosity, Water Absorption, Apparent Specific Gravity, and Bulk Density of Burned Refractory Products (A.S.T.M. Designation: C 20).[3]

(*j*) *Modulus of Rupture.*—Methods of Test for Cold Crushing Strength and Modulus of Rupture of Refractory Brick and Shapes (A.S.T.M. Designation: C 133).[3]

Standard Specifications for

REFRACTORIES FOR MODERATE DUTY STATIONARY BOILER SERVICE[1]

A.S.T.M. Designation: C 153 – 41

ADOPTED, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 153; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. (*a*) The purpose of these specifications is the selection of fireclay refractories for moderate duty boiler furnaces in which the temperature of the combustion chamber does not reach more than 2400 F. (1315 C.).

(*b*) These specifications are intended to cover only burned products made from fire clay by the usual processes of manufacture, and are not intended to include products other than those regularly sold as fireclay brick.

Zones of Highest Temperatures

2. The refractories to be used in the zones of highest temperatures, such as sidewalls, arches, doors, and jambs, shall be intermediate heat duty fireclay brick conforming to the requirements of type *H* (Section 5 (*a*)).

Zones of Lower Temperatures

3. The refractories to be used in the zones of lower temperatures, such as for backing up the brick in the zones of highest temperatures, and the walls beyond the first tube bank, shall be low heat duty fireclay brick conforming to the requirements of type *G* (Section 5 (*b*)).

REQUIREMENTS FOR THE TWO TYPES OF REFRACTORIES

Size and Warpage

4. The following permissible variations in size and warpage shall apply to each type of refractory listed in Section 5:

(*a*) *Size.*—When tested in accordance with the Methods of Test for Size and Bulk Density of Refractory Brick (A.S.T.M. Designation: C 134) of the American Society for Testing Materials,[3] the standard 9-in. fireclay brick shall not show a size variation of

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-8 on Refractories.

[2] Prior to adoption as standard, these specifications were published as tentative from 1940 to 1941. These specifications comprise a revision of the requirements for moderate duty refractories formerly contained in the Standard Specifications for Fireclay Brick for Stationary Boiler Service (C 64 – 39). Editorially revised in 1942.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

more than plus or minus 2 per cent. Tile or shapes shall not show a size variation of more than plus or minus 2 per cent on dimensions of 4 in. or over, and plus or minus 3 per cent on dimensions smaller than 4 in.

(*b*) *Warpage.*—Ninety-five per cent of the shapes shall not show a warpage greater than 1 per cent of the diagonal used in making the measurement.

Test Requirements

5. (*a*) ***Type H.***—Type *H* shall be an intermediate heat duty[4] fireclay brick conforming to the following requirements:

Refractoriness.—The brick shall be required to meet only *one* of the following requirements: Pyrometric cone equivalent not lower than cone No. 29, *or* not more than 3 per cent deformation in the 2460 F. (1350 C.) load test.

Permanent Linear Shrinkage.—When tested in accordance with schedule D (2460 F., 1350 C.), of the permanent linear change test, the brick shall not show a contraction of more than 1.5 per cent.

(*b*) ***Type G.***—Type *G* shall be a low heat duty[4] fireclay brick conforming to the following requirements:

Pyrometric Cone Equivalent.—The brick shall have a P.C.E. not lower than cone No. 19.

Modulus of Rupture.—The brick shall have a modulus of rupture of not less than 500 psi.

Retests

6. Because of variables resulting from sampling and the lack of perfect reproducibility in tests conducted by different laboratories, resampling and retesting may be carried out when requested by either the purchaser or the manufacturer. This may apply in instances where the first test result does not conform to the limits prescribed in these specifications. The final result to be used for each test shall be the average of the original and the retest figures.

NOTE.—A retest provision for variation in size is a part of that test method.

Methods of Testing

7. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials:

(*a*) *Size.*—Methods of Test for Size and Bulk Density of Refractory Brick (A.S.T.M. Designation: C 134).[3]

(*b*) *Warpage.*—Method of Test for Warpage of Refractory Brick and Tile (A.S.T.M. Designation: C 154).[3]

(*c*) *Pyrometric Cone Equivalent.*—Method of Test for Pyrometric Cone Equivalent (P.C.E.) of Refractory Materials (A.S.T.M. Designation: C 24).[3]

(*d*) *Load Test* (2460 F., 1350 C.).—Schedule No. 2 of the Method of Testing Fireclay Refractories Under Load at High Temperatures (A.S.T.M. Designation: C 16).[3]

(*e*) *Permanent Linear Shrinkage for Intermediate Heat Duty Brick* (2460 F., 1350 C.).—Schedule D of the Method of Test for Permanent Linear Change After Reheating of Refractory Brick (A.S.T.M. Designation: C 113).[3]

(*f*) *Modulus of Rupture.*—Methods of Test for Cold Crushing Strength and Modulus of Rupture of Refractory Brick and Shapes (A.S.T.M. Designation: C 133).[3]

[4] See the Classification of Fireclay Refractories (A.S.T.M. Designation C: 27), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for

REFRACTORIES FOR INCINERATORS[1]

A.S.T.M. Designation: C 106 - 41

ADOPTED, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 106; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. (*a*) The purpose of these specifications is the selection of fireclay refractories for service in municipal and industrial incinerators under general conditions of operation and furnace design.

(*b*) These specifications are intended to cover only burned products made from fire clay by the usual processes of manufacture, and are not intended to include products other than those regularly sold as fireclay brick.

Zones of Highest Temperatures

2. The refractories to be used in the zones of highest temperatures, such as the main body of the furnace, crown, and target wall, shall be high heat duty fireclay brick conforming to the requirements of type *A* (Section 5 (*a*)).

Lining of Chimneys

3. Because of various conditions which may be encountered in different installations, there are provided in this section of the specifications three types of refractories for the lining of chimneys. In order to select the type best suited for a particular installation, careful consideration should be given to the conditions that will exist. Unless the high heat duty brick, type *A*, or the low heat duty brick, type *G* is definitely specified, there shall be supplied for this section of the specifications the intermediate heat duty brick, type *F*.

REQUIREMENTS FOR THE VARIOUS TYPES OF REFRACTORIES

Size and Warpage

4. The following permissible variations in size and warpage shall apply to each type of refractory listed in Section 5:

(*a*) *Size.*—When tested in accordance with the Methods of Test for Size and Bulk Density of Refractory Brick (A.S.T.M. Designation: C 134) of the

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-8 on Refractories.

[2] Prior to their present adoption as standard, these specifications were published as tentative from 1934 to 1936, being revised in 1934. They were adopted in 1936, published as standard from 1936 to 1940, being revised in 1939, but withdrawn and republished as tentative from 1940 to 1941. Editorially revised in 1942.

American Society for Testing Materials,[3] the standard 9-in. fireclay brick shall not show a size variation of more than plus or minus 2 per cent. Tile or shapes shall not show a size variation of more than plus or minus 2 per cent on dimensions of 4 in. or over, and plus or minus 3 per cent on dimensions smaller than 4 in.

(*b*) *Warpage.*—Ninety-five per cent of the shapes shall not show a warpage greater than 1 per cent of the diagonal used in making the measurement.

Test Requirements

5. (*a*) ***Type A.***—Type *A* shall be a high heat duty[4] fireclay brick conforming to the following requirements:

Refractoriness.—The brick shall be required to meet only *one* of the following requirements: Pyrometric cone equivalent not lower than cone No. 31–32, *or* not more than 1.5 per cent deformation in the 2460 F. (1350 C.) load test.

Permanent Linear Shrinkage.—When tested in accordance with schedule B (2550 F., 1400 C.) of the permanent linear change test, the brick shall not show a contraction of more than 1.5 per cent.

Spalling.—When subjected to the panel spalling test (preheated at 2910 F., 1600 C.), the brick shall not show a loss greater than 15 per cent.

(*b*) ***Type F.***—Type *F* shall be an intermediate heat duty[4] fireclay brick conforming to the following requirements:

Refractoriness.—The brick shall be required to meet only *one* of the following requirements: Pyrometric cone equivalent not lower than cone No. 29, *or* not more than 3 per cent deformation in the 2460 F. (1350 C.) load test.

Modulus of Rupture.—The brick shall have a modulus of rupture of not less than 500 psi.

(*c*) ***Type G.***—Type *G* shall be a low heat duty[4] fireclay brick conforming to the following requirements:

Pyrometric Cone Equivalent.—The brick shall have a P.C.E. not lower than cone No. 19.

Modulus of Rupture.—The brick shall have a modulus of rupture of not less than 500 psi.

Retests

6. Because of variables resulting from sampling and the lack of perfect reproducibility in tests conducted by different laboratories, resampling and retesting may be carried out when requested by either the purchaser or the manufacturer. This may apply in instances where the first test result does not conform to the limits prescribed in these specifications. The final result to be used for each test shall be the average of the original and the retest figures.

NOTE.—A retest provision for variation in size is a part of that test method.

Methods of Testing

7. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials:

(*a*) *Size.*—Methods of Test for Size and Bulk Density of Refractory Brick (A.S.T.M. Designation: C 134).[3]

(*b*) *Warpage.*—Method of Test for Warpage of Refractory Brick and Tile (A.S.T.M. Designation: C 154).[3]

(*c*) *Pyrometric Cone Equivalent.*—Method of Test for Pyrometric Cone Equivalent (P.C.E.) of Refractory Materials (A.S.T.M. Designation: C 24).[3]

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[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

[4] See the Classification of Fireclay Refractories (A.S.T.M. Designation: C 27), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(*d*) *Load Test* (2460 F., 1350 C.).—Schedule No. 2 of the Method of Testing Fireclay Refractories Under Load at High Temperatures (A.S.T.M. Designation: C 16).[3]

(*e*) *Permanent Linear Shrinkage for High Heat Duty Brick* (2550 F., 1400 C.).—Schedule B of the Method of Test for Permanent Linear Change After Reheating of Refractory Brick (A.S.T.M. Designation: C 113).[3]

(*f*) *Panel Spalling Test for High Heat Duty Brick* (2910 F., 1600 C.).—Method of Panel Test for Resistance to Thermal and Structural Spalling of High Heat Duty Fireclay Brick (A.S.T.M. Designation: C 107).[3]

(*g*) *Modulus of Rupture.*—Methods of Test for Cold Crushing Strength and Modulus of Rupture of Refractory Brick and Shapes (A.S.T.M. Designation: C 133).[3]

Standard Specifications for

REFRACTORIES FOR MALLEABLE IRON FURNACES WITH REMOVABLE BUNGS, AND FOR ANNEALING OVENS[1]

A.S.T.M. Designation: C 63 – 41

ADOPTED, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 63; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. (*a*) The purpose of these specifications is the selection of fireclay refractories for service in malleable furnaces and annealing ovens under general conditions of operation and furnace design.

(*b*) These specifications are intended to cover only burned products made from fire clay by the usual processes of manufacture, and are not intended to include products other than those regularly sold as fireclay brick.

Malleable Furnace Bungs

2. The refractories to be used in the bungs shall be a special type of fireclay brick conforming to the requirements of type *I* (Section 8 (*a*)).

Furnace Sidewalls and Bridgewalls

3. The refractories to be used in malleable furnace sidewalls and bridgewalls shall be high heat duty fireclay brick conforming to the requirements of type *J* (Section 8 (*b*)).

Lining of Fire boxes and Lower Portion of Stack

4. The refractories to be used in lining the fireboxes of malleable iron furnaces and of annealing ovens, and the lower portion of the stack, shall be high heat duty fireclay brick conforming to the requirements of type *K* (Section 8 (*c*)).

Upper Stack Lining

5. The refractories to be used in lining the upper portion of the stack shall be intermediate heat duty fireclay brick conforming to the requirements of type *H* (Section 8 (*d*)).

Annealing Oven Chamber

6. The refractories to be used in constructing the annealing oven chamber shall be low heat duty fireclay brick conforming to the requirements of type *L* (Section 8 (*e*)).

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-8 on Refractories.

[2] Prior to their present adoption as standard, these specifications were published as tentative from 1927 to 1928. They were adopted in 1928, published as standard from 1928 to 1934, but withdrawn and republished as tentative from 1934 to 1936. They were adopted in 1936, published as standard from 1936 to 1940, being revised in 1939, but withdrawn and republished as tentative from 1940 to 1941. Editorially revised in 1942.

REQUIREMENTS FOR THE VARIOUS TYPES OF REFRACTORIES

Size and Warpage

7. The following permissible variations in size and warpage shall apply to each type of refractory listed in Section 8:

(*a*) *Size.*—When tested in accordance with the Methods of Test for Size and Bulk Density of Refractory Brick (A.S.T.M. Designation: C 134) of the American Society for Testing Materials,[3] the standard 9-in. fireclay brick shall not show a size variation of more than plus or minus 2 per cent. Tile or shapes shall not show a size variation of more than plus or minus 2 per cent on dimensions of 4 in. or over, and plus or minus 3 per cent on dimensions smaller than 4 in.

(*b*) *Warpage.*—Ninety-five per cent of the shapes shall not show a warpage greater than 1 per cent of the diagonal used in making the measurement.

Test Requirements

8. (*a*) ***Type I.***—Type *I* shall be a special type of fireclay refractory known as bung brick, conforming to the following requirements:

Pyrometric Cone Equivalent.—The brick shall have a P.C.E. not lower than cone No. 32.

Spalling.—When subjected to the panel spalling test (preheated at 2910 F., 1600 C.), the brick shall not show a loss greater than 7.5 per cent.

Modulus of Rupture.—The brick shall have a modulus of rupture of not less than 200 psi.

(*b*) ***Type J.***—Type *J* shall be a high heat duty[4] fireclay brick conforming to the following requirements:

Pyrometric Cone Equivalent.—The brick shall have a P.C.E. not lower than cone No. 32.

Permanent Linear Shrinkage.—When tested in accordance with schedule B (2550 F., 1400 C.) of the permanent linear change test, the brick shall not show a contraction of more than 1.5 per cent.

(*c*) ***Type K.***—Type *K* shall be a high heat duty[4] fireclay brick conforming to the following requirements:

Refractoriness.—The brick shall be required to meet only *one* of the following requirements: Pyrometric cone equivalent not lower than cone No. 31–32, *or* not more than 1.5 per cent deformation in the 2460 F. (1350 C.) load test.

Permanent Linear Shrinkage.—When tested in accordance with schedule B (2550 F., 1400 C.) of the permanent linear change test, the brick shall not show a contraction of more than 1.5 per cent.

(*d*) ***Type H.***—Type *H* shall be an intermediate heat duty[4] fireclay brick conforming to the following requirements:

Refractoriness.—The brick shall be required to meet only *one* of the following requirements: Pyrometric cone equivalent not lower than cone No. 29, *or* not more than 3 per cent deformation in the 2460 F. (1350 C.) load test.

Permanent Linear Shrinkage.—When tested in accordance with schedule D (2460 F., 1350 C.) of the permanent linear change test, the brick shall not show a contraction of more than 1.5 per cent.

(*e*) ***Type L.***—Type *L* shall be a low heat duty[4] fireclay brick conforming to the following requirements:

Pyrometric Cone Equivalent.—The brick shall have a P.C.E. not lower than cone No. 19.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

[4] See the Classification of Fireclay Refractories (A.S.T.M. Designation: C 27), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Permanent Linear Shrinkage.—When tested in accordance with schedule A (2190 F., 1200 C.) of the permanent linear change test, the brick shall not show a contraction of more than 1.5 per cent.

Retests

9. Because of variables resulting from sampling and the lack of perfect reproducibility in tests conducted by different laboratories, resampling and retesting may be carried out when requested by either the purchaser or the manufacturer. This may apply in instances where the first test result does not conform to the limits prescribed in these specifications. The final result to be used for each test shall be the average of the original and the retest figures.

NOTE.—A retest provision for variation in size is a part of that test method.

Methods of Testing

10. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials:

(*a*) *Size.*—Methods of Test for Size and Bulk Density of Refractory Brick (A.S.T.M. Designation: C 134).[3]

(*b*) *Warpage.*—Method of Test for Warpage of Refractory Brick and Tile (A.S.T.M. Designation: C 154).[3]

(*c*) *Pyrometric Cone Equivalent.*—Method of Test for Pyrometric Cone Equivalent (P.C.E.) of Refractory Materials (A.S.T.M. Designation: C 24).[3]

(*d*) *Load Test* (2460 F., 1350 C.).—Schedule No. 2 of the Method of Testing Fireclay Refractories Under Load at High Temperatures (A.S.T.M. Designation: C 16).[3]

(*e*) *Permanent Linear Shrinkage for Low Heat Duty Brick* (2190 F., 1200 C.).—Schedule A of the Method of Test for Permanent Linear Change After Reheating of Refractory Brick (A.S.T.M. Designation: C 113).[3]

(*f*) *Permanent Linear Shrinkage for Intermediate Heat Duty Brick* (2460 F., 1350 C.).—Schedule D of the Method of Test for Permanent Linear Change After Reheating of Refractory Brick (A.S.T.M. Designation: C 113).[3]

(*g*) *Permanent Linear Shrinkage for High Heat Duty Brick* (2550 F., 1400 C.).—Schedule B of the Method of Test for Permanent Linear Change After Reheating of Refractory Brick (A.S.T.M. Designation: C 113).[3]

(*h*) *Panel Spalling Test for High Heat Duty Brick* (2910 F., 1600 C.).—Method of Panel Test for Resistance to Thermal and Structural Spalling of High Heat Duty Fireclay Brick (A.S.T.M. Designation: C 107).[3]

(*i*) *Modulus of Rupture.*—Methods of Test for Cold Crushing Strength and Modulus of Rupture of Refractory Brick and Shapes (A.S.T.M. Designation: C 133).[3]

Standard Specifications for

FIRECLAY PLASTIC REFRACTORIES FOR BOILER AND INCINERATOR SERVICES[1]

A.S.T.M. Designation: C 176 – 44

ADOPTED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 176; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. (*a*) These specifications cover fireclay plastic refractories for use in boiler furnaces and municipal or industrial incinerators operated under general conditions of service.

(*b*) In addition to high heat duty plastic refractories for average service conditions, super duty plastic refractories are covered, because for some furnaces the design and operation may justify the use of the latter. The use of super duty plastic refractories may be desirable in the zone of highest temperature and when severe spalling or slagging is encountered, and especially when both slagging and spalling may occur to an unusual extent. Unless otherwise specified, high heat duty plastic refractories shall be supplied.

High Heat Duty Plastic Refractories Required for Average Service Conditions

2. (*a*) *Water Content.*—The plastic refractory as received shall contain not more than 15 per cent of water calculated on the basis of the wet weight.

(*b*) *Workability Index.* — The workability index of the plastic refractory as received shall be not less than 15 per cent nor more than 35 per cent deformation. The test specimen shall stand three tamps of the test weight without crumbling.

(*c*) *Pyrometric Cone Equivalent.*—The P.C.E. of the plastic refractory shall be not lower than cone No. 31.

(*d*) *Drying-and-Firing Shrinkage.*—The total drying-and-firing (2550 F., 1400 C.) linear shrinkage of the plastic refractory shall be not more than 4 per cent of the original length.

(*e*) *Panel Spalling Loss.*—The panel spalling (preheated at 2910 F., 1600 C.) loss of the plastic refractory shall be not more than 15 per cent.

Super Duty Plastic Refractories for Use in the Zone of Highest Temperature and Where Severe Spalling or Slagging Occurs

3. (*a*) *Water Content.*—The plastic refractory as received shall contain not

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-8 on Refractories.

[2] Prior to adoption as standard, these specifications were published as tentative from 1942 to 1944.

more than 15 per cent of water calculated on the basis of the wet weight.

(*b*) *Workability Index.* — The workability index of the plastic refractory as received shall be not less than 15 per cent nor more than 35 per cent deformation. The test specimen shall stand at least three tamps of the test weight without crumbling.

(*c*) *Pyrometric Cone Equivalent.*—The P.C.E. of the plastic refractory shall be not lower than cone No. 32½.

(*d*) *Drying-and-Firing Shrinkage.*—The total drying-and-firing (2910 F., 1600 C.) linear shrinkage of the plastic refractory shall be not more than 2.5 per cent of the original length.

(*e*) *Panel Spalling Loss.*—The panel spalling (preheated at 3000 F., 1650 C.) loss of the plastic refractory shall be not more than 5 per cent.

Retests

4. Because of the variables resulting from sampling and the lack of perfect reproducibility in tests conducted by different laboratories, the plastic refractory may be resampled and retested when requested by either the manufacturer or the purchaser. This may apply in instances where the first test result does not conform to the limits prescribed in these specifications. The final result to be used for each test shall be the average of the original and the retest values.

Methods of Testing

5. The properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(*a*) *Water Content.*—Methods of Test for Sieve Analysis and Water Content of Refractory Materials (A.S.T.M. Designation: C 92).[3]

(*b*) *Workability Index.*—Method of Test for Workability Index of Fireclay Plastic Refractories (A.S.T.M. Designation: C 181).[3]

(*c*) *Pyrometric Cone Equivalent.*—Method of Test for Pyrometric Cone Equivalent (P.C.E.) of Refractory Materials (A.S.T.M. Designation: C 24).[3]

(*d*) *Drying and Firing Shrinkage.*—Method of Test for Combined Drying and Firing Shrinkage of Fireclay Plastic Refractories (A.S.T.M. Designation: C 179).[3]

(*e*) *Panel Spalling Loss.*—Method of Panel Test for Resistance to Thermal and Structural Spalling of Fireclay Plastic Refractories (A.S.T.M. Designation: C 180).[3]

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[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for

GROUND FIRE CLAY AS A MORTAR FOR LAYING-UP FIRECLAY BRICK[1]

A.S.T.M. Designation: C 105 - 41

ADOPTED, 1936; REVISED, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 105; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover ground fire clays to be used in laying-up fireclay brick.

Quality

2. The material shall be sufficiently free from grit and of such quality and plasticity that it will spread satisfactorily with a trowel when tempered with adequate water.

Grades

3. There are two grades of ground fire clay, depending upon particle size, as follows:

	Designation
Commercial grade	C
Fine grade	F

Particle Size

4. (*a*) *Commercial Grade.*—Not more than 5 per cent shall be retained on a No. 20 A.S.T.M. sieve[3] (equivalent to mesh No. 20 of the Tyler standard series), and not more than 0.5 per cent shall be retained on a No. 16 A.S.T.M. sieve (equivalent to mesh No. 14 of the Tyler standard series).

(*b*) *Fine Grade.*—Not more than 5 per cent shall be retained on a No. 30 A.S.T.M. sieve[3] (equivalent to mesh No. 28 of the Tyler standard series), and not more than 0.5 per cent shall be retained on a No. 20 A.S.T.M. sieve (equivalent to mesh No. 20 of the Tyler standard series).

Classes

5. There shall be four classes of ground fire clay depending upon the grade of refractories with which they are to be used. These classes shall conform to the following requirements as to Pyrometric Cone Equivalent:

Class[4]	Pyrometric Cone Equivalent, Not Lower Than
Super duty	No. 31
High heat duty	No. 28
Intermediate heat duty	No. 26
Low heat duty	No. 16

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-8 on Refractories.

[2] Prior to adoption as standard, these specifications were published as tentative from 1934 to 1936. Editorially revised in 1943.

[3] Detailed requirements for these sieves are given in the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

[4] Each class shall be suffixed by the designating letter C (commercial grade) or F (fine grade) depending upon the particle size required.

A "tentative revision" of this standard appears in the Tentative Revisions of Standards section at the back of this book.

Sampling

6. (*a*) *Containers.*—When ground fire clay is shipped in containers, a sample shall be taken as follows:

If the shipment consists of 100 containers or fraction thereof, the contents of one container, selected at random, shall be dumped and quartered to provide a 5-lb. sample for the laboratory. If the shipment consists of more than 100 containers, the number of containers to be sampled shall be as follows:

Number of Containers in Shipment	Number of Containers Selected for Sampling
Over 100 but less than 300	2
300 but less than 600	3
600 but less than 1000	4

In these cases, the several 5-lb. samples resulting from quartering shall be combined and either quartered or riffled to provide a 5-lb. sample for the laboratory.

(*b*) *Bulk.*—When ground fire clay is shipped in bulk, the sample shall be so taken that it will represent an average of all parts of the shipment from top to bottom and shall not contain a disproportionate share of the top and bottom layers. For a 30-ton unit, no less than 10 samples of approximately 25 lb. each shall be taken from different parts of the car or truck by digging holes spaced at equal intervals. If the shipment is more than 30 tons, a sample proportionately larger shall be taken. The combined samples shall be thoroughly mixed and quartered or riffled to provide a 5-lb. sample for the laboratory.

It shall be permissible to obtain the sample during unloading by taking a small scoopful of clay at regular intervals. One scoopful of clay, weighing approximately 1½ lb., shall be taken for each ton of material. In no case, however, shall the combined samples weigh less than 50 lb. The combined samples shall be thoroughly mixed and quartered or riffled to provide a 5-lb. sample for the laboratory.

Methods of Testing

7. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials:

(*a*) *Particle Size.*—The procedure for wet sieve analysis as described in Section 5 of the Methods of Test for Sieve Analysis and Water Content of Refractory Materials (A.S.T.M. Designation: C 92).[4]

(*b*) *Pyrometric Cone Equivalent.*—Method of Test for Pyrometric Cone Equivalent (P.C.E.) of Refractory Materials (A.S.T.M. Designation: C 24).[4]

[4] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Classification of

FIRECLAY REFRACTORIES[1]

A.S.T.M. Designation: C 27 - 41

ADOPTED, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 27; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This classification is intended principally to group fireclay brick according to their resistance to heat.

Super Duty Fireclay Brick

2. *Super Duty Fireclay Brick.*—A fireclay brick having a pyrometric cone equivalent not lower than cone No. 33 on the fired product, not more than 1 per cent linear shrinkage in the permanent linear change test, schedule C (2910 F., 1600 C.), and not more than 4 per cent loss in the panel spalling test (preheated at 3000 F., 1650 C.).

High Heat Duty Fireclay Brick

3. *High Heat Duty Fireclay Brick.*—A fireclay brick conforming to only *one* of the following requirements: Pyrometric cone equivalent not lower than cone No. 31-32, *or* not more than 1.5 per cent deformation in the 2460 F. (1350 C.) load test.

Intermediate Heat Duty Fireclay Brick

4. *Intermediate Heat Duty Fireclay Brick.*—A fireclay brick conforming to only *one* of the following requirements: Pyrometric cone equivalent not lower than cone No. 29, *or* not more than 3 per cent deformation in the 2460 F. (1350 C.) load test.

Low Heat Duty Fireclay Brick

5. *Low Heat Duty Fireclay Brick.*—A fireclay brick having a pyrometric cone equivalent not lower than cone No. 19.

Alumina-Diaspore Fireclay Brick

6. (*a*) *50 per cent Alumina-Diaspore Fireclay Brick.*—Refractory brick having diaspore or nodular fire clay as an essential original ingredient, an alumina content of 50 ± 2.5 per cent, and a pyrometric cone equivalent not lower than cone No. 34.

(*b*) *60 per cent Alumina-Diaspore Fireclay Brick.*—Refractory brick having diaspore or nodular fire clay as an essential original ingredient, an alumina content of 60 ± 2.5 per cent, and a pyrometric cone equivalent not lower than cone No. 35.

[1] Under the standardization procedure of the Society, this classification is under the jurisdiction of the A.S.T.M. Committee C-8 on Refractories.

[2] Prior to its present adoption as standard, this classification was published as tentative from 1919 to 1920. It was adopted in 1920, published as standard from 1920 to 1940, being revised in 1928, 1935, 1937, and 1939, but withdrawn and republished as tentative from 1940 to 1941. Editorially revised in 1942.

(*c*) *70 per cent Alumina-Diaspore Fireclay Brick.*—Refractory brick having diaspore as an essential original ingredient, an alumina content of 70 ± 2.5 per cent, and a pyrometric cone equivalent not lower than cone No. 36.

NOTE.—Definitions of diaspore clay and nodular fire clay appear in the Definitions of Terms Relating to Refractories (A.S.T.M. Designation: C 71) of the American Society for Testing Materials.[2]

Methods of Testing

7. The properties enumerated in this classification shall be determined in accordance with the following methods of test of the American Society for Testing Materials:

(*a*) *Pyrometric Cone Equivalent.*—Method of Test for Pyrometric Cone Equivalent (P.C.E.) of Refractory Materials (A.S.T.M. Designation: C 24).[3]

(*b*) *Permanent Linear Shrinkage for Super Duty Brick* (2910 F., 1600 C.).—Schedule C of the Method of Test for Permanent Linear Change After Reheating of Refractory Brick (A.S.T.M. Designation: C 113).[3]

(*c*) *Panel Spalling Test for Super Duty Brick* (3000 F., 1650 C.).—Method of Panel Test for Resistance to Thermal and Structural Spalling of Super Duty Fireclay Brick (A.S.T.M. Designation: C 122).[3]

(*d*) *Load Test* (2460 F., 1350 C.).—Schedule No. 2 of the Method of Testing Fireclay Refractories Under Load at High Temperatures (A.S.T.M. Designation: C 16).[3]

(*e*) *Alumina Content.*—Methods of Chemical Analysis of Refractory Materials (A.S.T.M. Designation: C 18).[3]

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[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Methods of

CHEMICAL ANALYSIS OF REFRACTORY MATERIALS[1]

A.S.T.M. Designation: C 18 – 41

ADOPTED, 1939; REVISED, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 18; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. (*a*) These methods cover procedures for the chemical analysis of fireclay, silica, high-alumina, and magnesite refractories, and of chrome ore and chrome refractories.

(*b*) The analytical procedures appear in the following order:

	Section
Fireclay Refractories:	
Moisture	7
Loss on Ignition	8
Silica	9
Iron, Aluminum, and Titanium Oxides	10
Iron Oxide	11
Titania	12
Alumina	13
Lime	14
Magnesia	15
Alkalies	16
Silica Refractories:	
Moisture	18
Loss on Ignition	19
Silica	20
Iron and Aluminum Oxides	21
Iron Oxide	22
Titania	23
Alumina	24
Lime	25
Magnesia	26
Alkalies	27
High-Alumina Refractories:	
Moisture	29
Loss on Ignition	30
Silica	31
Separation of Al_2O_3 and P_2O_5 from Fe_2O_3, TiO_2, CaO, and MgO	32
Iron Oxide	33
Titania	34
Phosphorus Oxide	35
Alumina	36
Lime	37
Magnesia	38
Alkalies	39
Magnesite Refractories:	
Moisture	42
Loss on Ignition	43
Silica	44
Iron and Aluminum Oxides	45
Iron Oxides	46
Alumina	47
Lime	48
Magnesia	49
Manganese as MnO	50
Chrome Ore and Chrome Refractories:	
Moisture	53
Loss on Ignition	54
Chromium Oxide	55
Titania and Ferrous Oxide	56
Titania	57
Ferrous Oxide	58
Silica	59
Alumina	60
Lime	61
Manganese as MnO	62
Magnesia	63

NOTE.—These methods have been compiled as standard procedures for use in referee analyses. These methods, however, when the determination of iron oxide as Fe_2O_3 is involved, are not intended to preclude the use of other procedures that give results within the permissible varia-

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee C-8 on Refractories.

[2] Prior to their present adoption as standard, these methods were published as tentative from 1917 to 1920, being revised in 1918. They were adopted in 1920, published as standard from 1920 to 1937, being revised in 1935, but were withdrawn and published as tentative from 1937 to 1939.

tions. For the sake of uniformity the classical Zimmerman-Reinhardt procedure is specified for the determination of iron oxide. It is recognized that numerous other procedures are equally accurate and often more convenient. The other procedures commonly in use include reduction of an oxidized solution with zinc or other metal, and titration with standard $KMnO_4$ or $K_2Cr_2O_7$, as well as titration with a standard solution of titanous chloride in an oxidized solution. These procedures shall be considered acceptable provided the analyst has obtained results by his special procedure which check with the Zimmerman-Reinhardt procedure within the limits specified in Section 6. It is suggested that the National Bureau of Standards' standard samples be used for checking the accuracy of procedures.

It will be understood that the making of a complete analysis of a refractory material is a difficult procedure requiring a wide knowledge of the chemistry involved in the operations and a thorough training in carrying out the work. A skilled analyst of good training is therefore required to do the work. The descriptions here given cover the vital points of procedure, but frequent reference in regard to the details of the various manipulations should be made to "Applied Inorganic Analysis" by Hillebrand and Lundell[3] and to similar publications. Particularly in the determination of alumina, reference should be made to *Scientific Paper No. 286* of the National Bureau of Standards.[4]

Reagents and Special Solutions Required

2. The lists in Paragraphs (*a*) to (*n*) include those reagents and special solutions common to two or more of the analytical procedures. Other special reagents and solutions required will be found listed with the particular method in which they are prescribed.

(*a*) *Concentrated Acids and Ammonium Hydroxide.*—Concentrated c.p. acids and ammonium hydroxide of approximately the following specific gravities or concentrations will be required:

Hydrochloric acid, HCl	1.19 sp. gr.
Nitric acid, HNO_3	1.42 sp. gr.
Sulfuric acid, H_2SO_4	1.84 sp. gr.
Hydrofluoric acid, HF	40 per cent
Perchloric acid, $HClO_4$	60 to 70 per cent, c. p.[a]
Sulfurous acid	6 per cent solution[b]
Ammonium hydroxide, NH_4OH	0.90 sp. gr.

[a] Lower purity varieties may contain aluminum oxide, Al_2O_3, as an impurity.
[b] As supplied by reagent manufacturers.

(*b*) *Diluted Acids and Ammonium Hydroxide.*—The diluted acids and ammonium hydroxide referred to are of varying percentages by volume. They shall be made up by mixing proportional volumes of the concentrated reagent and water. The diluted sulfuric acid mixtures shall be made up by slowly stirring the acid into the water. These diluted acids and ammonium hydroxide are designated in the methods as (1:4), (1:9), etc., except very diluted solutions which are referred to by the percentage of reagent added. The designation in parentheses indicates the ratio of the volume of the concentrated reagent to the volume of water; for example, H_2SO_4 (1:9) contains 10 per cent by volume of H_2SO_4 (sp. gr. 1.84). The following will be required:

	Percentage by Volume
Hydrochloric acid	50 20
Sulfuric acid	50 10 5
Nitric acid	30 3
Ammonium hydroxide	50 5

(*c*) *Ammonium Chloride (2 per cent).*—Dissolve 2 g. of NH_4Cl in 100 ml. of water.

(*d*) *Ammonium Oxalate (Saturated Solution).*—Dissolve 4 g. of $(NH_4)_2C_2O_4$ in 100 ml. of water.

(*e*) *Standard Potassium Permanganate (0.1 N).*—Dissolve 3.25 g. of $KMnO_4$ in 1000 ml. of water. Standardize against the National Bureau of Standards' standard sample 40*c* of sodium oxalate.

[3] W. F. Hillebrand and G. E. F. Lundell, "Applied Inorganic Analysis," Wiley and Son, New York City (1929).
[4] W. Blum, "Determination of Alumina as Oxide," National Bureau of Standards *Scientific Paper No. 286.*

(*f*) *Standard Potassium Permanganate* (*0.04 N*).—Dissolve 2.5 g. of $KMnO_4$ in water and make up to 2 liters. Allow to stand for a week, filter through an asbestos mat, and keep in a dark place. Standardize against the National Bureau of Standards' standard sample 40*c* of sodium oxalate.

(*g*) *Standard Titania Solution.*—Weigh out 0.05 g. of pure calcined TiO_2. Fuse with 10 g. of $K_2S_2O_7$ in a clean platinum crucible, keeping the temperature as low as possible to maintain fluidity. Cool, and dissolve in about 300 ml. of diluted H_2SO_4 (1:5). Cool, transfer to a 500-ml. volumetric flask, dilute to the mark with water, and mix thoroughly. To standardize the solution, take two 50-ml. portions in 400-ml. beakers, dilute, boil, and precipitate with NH_4OH. Filter, and wash with hot water. Place the papers in the original beakers, add 15 ml. of HCl, stir to macerate the paper, dilute, and precipitate again with NH_4OH. Filter, and wash with hot water until free of alkali salts. Ignite carefully, blast, and weigh. From the weight determined, calculate the strength of the solution.

(*h*) *Standard Sodium Arsenite Solution.*—Dissolve 0.908 g. of pure arsenious oxide, As_2O_3, in a small amount of hot Na_2CO_3 solution, cool, filter, and dilute to 1 liter. Standardize against a steel of known manganese content.

(*i*) *Stannous Chloride Solution.*—Dissolve 50 g. of $SnCl_2$ in 100 ml. of HCl and dilute to 1000 ml. Keep a few pieces of metallic tin in the bottle.

(*j*) *Mercuric Chloride Solution.*—Prepare a saturated solution of $HgCl_2$.

(*k*) *Manganese Sulfate Solution.*[4a]—Dissolve 70 g. of crystalline $MnSO_4$ in 500 ml. of water. Add 140 ml. of phosphoric acid, H_3PO_4, (sp. gr. 1.7) and 130 ml. of H_2SO_4 (sp. gr. 1.84). Dilute to 1 liter.

(*l*) *Hydrogen Peroxide* (*30 per cent*).

(*m*) *Diammonium Phosphate Solution.*—Dissolve 10 g. of $(NH_4)_2HPO_4$ in 100 ml. of water.

(*n*) *Chloroplatinic Acid Solution* (*10 per cent*).

(*o*) *Ethyl Alcohol* (80 *per cent*).—Prepare a solution containing 80 per cent by volume of ethyl alcohol in water.

(*p*) *Ethyl Alcohol* (*Absolute*).—Certain commercial brands of denatured absolute alcohol are satisfactory as well as being considerably less expensive than the c.p. absolute alcohol.

Preparation of Samples for Analysis

3. The sample, carefully obtained by one of the approved methods of sampling, shall be crushed in a small jaw or roll-type crusher with hardened tool-steel faces to pass a No. 8 (2380-micron) sieve.[5] The sample shall then be crushed to pass a No. 20 (840-micron) sieve,[5] mixed, and quartered to about 50 g. This 50-g. sample shall then be ground so that it will all pass a No. 100 (149-micron) sieve,[5] unless otherwise specified, mixed thoroughly, and placed in a container which will insure freedom from contamination. Fine grinding shall be done in an agate mortar, either by hand or by a mechanical sample grinder of the McKenna, Carling, Fisher, or similar type so constructed as to prevent the introduction of impurity.

Statement of Analysis[5a]

4. Moisture shall be determined on the sample in its ordinary air-dried condition. All other percentage compositions shall be determined on moisture-free samples and reported, accordingly, on a moisture-free basis. The drying temperature recommended for all mois-

[4a] This paragraph was editorially revised in 1943.

[5] Detailed requirements for these sieves are given in the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

[5a] This section was editorially revised in 1942.

ture determinations is 105 to 110 C., although it is recognized that in the case of certain raw fire clays a temperature as high as 140 C. may be required for complete removal of all hygroscopic moisture.[6] Whenever a sample is weighed out for any determination other than moisture, it shall be moisture-free. If preferred, the sample may be dried in a weighing bottle from which the required samples shall be weighed out.

Blank Determinations

5. Blank determinations on the reagents shall be made for each constituent in the refractory and this blank deducted in each case. For the determination of the silica blank, approximately 0.25 g. of Al_2O_3 should be added as pure aluminum chloride.

Reproducibility of Results

6. In all cases, check determinations shall be made, and the results shall be redetermined if satisfactory checks are not obtained. Results shall be considered satisfactory if the differences between check determinations do not exceed the following limits:

	Permissible Variations Between Check Determinations, max., per cent
For silica or other constituent amounting to 30 per cent[a] or over	0.3
For alumina or other constituent amounting to 10 to 30 per cent[a]	0.2
For any other constituent amounting to under 10 per cent[a]	0.1

[a] These figures are stated in terms of the whole sample as 100 per cent.

FIRECLAY REFRACTORIES

Moisture

7. Weigh 1.00 g. of the sample and heat to constant weight at a temperature not under 105 nor over 110 C. Record the loss in weight as moisture.

Loss on Ignition

8. Weigh 1.000 g. of the moisture-free (105 to 110 C.) sample and heat to constant weight over a blast lamp, or in an electric muffle furnace, at 900 to 1000 C. Record the loss in weight as the ignition loss.

Silica

9. Weigh 0.5000 g. of the moisture-free (105 to 110 C.) sample into a platinum crucible containing about 5 g. of powdered anhydrous Na_2CO_3 and mix well with a platinum wire. Cover the mixture with a little more Na_2CO_3. Heat gradually to the full heat of a good burner (1000 to 1100 C.) maintained for about 1 hr. until complete solution is obtained. Place the crucible cover on a triangle, and when the melt has partially cooled pour it on the lid (Note). When cool, place the crucible and lid in a 150-ml. beaker, placing the button on a watch glass above the beaker. Add 30 ml. of HCl (1:1). When solution is complete wash off the crucible and lid with HCl (1:4), taking care to remove all SiO_2. Place the button in the solution. Transfer the contents of the beaker to an evaporating dish and evaporate to dryness on a steam bath. Bake for 1 hr. at 110 C. Add 20 to 30 ml. of HCl (1:1) and 50 ml. of hot water. When all salts have been dissolved, allow to settle for several minutes and then filter through a No. 40 Whatman paper or equivalent. Wash the SiO_2 three times by decantation using 20 to 30-ml. portions of first hot water, then HCl (1:1), then hot water again. Transfer the precipitate to the filter paper, removing all SiO_2 from the dish with a policeman. Wash the paper and precipitate with hot water until free from salt. To recover the small amount of

[6] See National Bureau of Standards' Certificates of Analysis of Standard Samples of Fire Clays, Nos. 97 and 98, August 31, 1931.

SiO_2 remaining in the filtrate, evaporate to dryness, using the same procedure for baking and filtering as before. Combine the two precipitates, place in a platinum crucible, and burn off the paper carefully to prevent any loss of SiO_2. Ignite the sample to constant weight at 1100 to 1200 C. (15 to 20 min. is usually sufficient), cool in a desiccator, and weigh. Moisten the residue with several milliliters of water, add 10 ml. of HF and 3 or 4 drops of H_2SO_4. Evaporate the solution to dryness, ignite carefully to prevent decrepitation, and blast for several minutes at 1100 C. Cool the crucible in a desiccator, weigh, and repeat blasting to constant weight. The loss in weight from the original silica residue represents the SiO_2 content, except for that part of the SiO_2 which is later recovered from alumina, etc.

NOTE.—Another scheme to aid in subsequent solution of the fused melt is to rotate the crucible as it cools, spreading the mass up the side walls.

Iron, Aluminum, and Titanium Oxides

10. Fuse the residue with 1 g. of fused $K_2S_2O_7$ or $Na_2S_2O_7$, dissolve in a small amount of water, and add to the filtrate from the silica determination (Section 9). Add 5 g. of NH_4Cl and 3 drops of methyl red solution (0.1 per cent). Heat the solution almost to boiling, and add slowly NH_4OH (1:1) until the indicator has changed to a yellow color. Boil for several minutes to remove the excess ammonia. Allow to settle for 30 min. and decant through a No. 41 Whatman filter paper or equivalent, transferring the precipitate to the paper and washing the beaker and paper several times with a warm NH_4Cl solution (2 per cent). Reserve the filtrate, *A*, for the determination of CaO and MgO (Section 14). Return the precipitate to the original beaker, add 50 ml. of hot water and 10 ml. of HCl (sp. gr. 1.19). Stir until the precipitate is dissolved and the paper is well macerated. Dilute to about 200 ml. with hot water, precipitate and filter as before. Combine this filtrate *B* with filtrate *A*. Wash the paper and precipitate with a warm NH_4Cl solution (2 per cent). Place the precipitate in a weighed platinum crucible and ignite. Continue the ignition at 1200 C. to constant weight (15 to 20 min. is usually sufficient). Cool in a desiccator, and weigh with the crucible covered with the lid. The R_2O_3 consists of the Al_2O_3, TiO_2, and Fe_2O_3 present in the sample. In addition there may be small amounts of P_2O_5, ZrO_2, V_2O_5 and Cr_2O_3.

Iron Oxide

11. (*a*) *Method A: Fe_2O_3 Determined on R_2O_3 Sample.*—Heat the R_2O_3 precipitate (Note) obtained in the determination of iron, aluminum, and titanium oxides (Section 10) with fused $K_2S_2O_7$ or $Na_2S_2O_7$ until solution is complete. Dissolve the fusion in 50 ml. of H_2SO_4 (1:9) and evaporate to fumes. Cool, dilute with water, and filter off the SiO_2, washing with hot water. Reserve the filtrate for the determination of Fe_2O_3 and TiO_2. Ignite the SiO_2 in a platinum crucible and weigh. Treat the precipitate with 5 ml. of HF and 2 or 3 drops of H_2SO_4. Evaporate to dryness, ignite, and weigh. The loss in weight represents extra SiO_2 which should be added to that determined previously and also deducted from the weight of the R_2O_3 precipitate. Evaporate the filtrate obtained in correcting the R_2O_3 precipitate for SiO_2 to about 75 ml. Cool, and dilute to 100 ml. in a volumetric flask. Reserve 25 ml. for the determination of TiO_2 (Section 12). To the remainder, add 25 ml. of HCl (1:1) and heat to boiling. Reduce the iron by adding $SnCl_2$ solution drop by drop from a pipette with constant swirling of the beaker until the solution is

colorless. Then add one drop in excess. Cool quickly in running water, then add at one stroke 15 ml. of saturated $HgCl_2$ solution. Allow to stand for 3 min., then transfer with washing to a 1000-ml. beaker containing 300 ml. of cold distilled water and 25 ml. of $MnSO_4$ solution. Titrate with standard 0.04 *N* $KMnO_4$, added very slowly while stirring constantly, until a permanent pink end point is obtained.

NOTE.—Instead of fusing directly in the platinum crucible in which the R_2O_3 was ignited, the precipitate may be brushed into a porcelain crucible and then fused with $K_2S_2O_7$ or $Na_2S_2O_7$. This avoids loss of platinum by the action of the pyrosulfate, and no platinum is present in the filtrate to interfere with the iron determination.

(*b*) *Method B: Fe_2O_3 Determined on a Separate Sample.*—Weigh 1.00 g. of the finely ground, moisture-free (105 to 110 C.) sample into a platinum crucible, add 10 drops perchloric acid ($HClO_4$) and 20 ml. of HF, and heat almost to dryness on a hot plate. Add 5 to 10 ml. of $HClO_4$ and heat until residue has dissolved (Note). Cool, place crucible in a 400-ml. beaker, add 100 ml. of water, and heat to boiling. Any residue present, other than SiO_2, should be filtered off and fused with $K_2S_2O_7$ or $Na_2S_2O_7$ in a porcelain crucible and added to the main solution.

NOTE.—Decomposition of raw fire clays and some fired refractories can be effected equally as well by substituting an equal volume of H_2SO_4 (1:1) for the $HClO_4$. In this case, heat to fumes once, cool, dilute, filter, and fuse any residue remaining undissolved.

(*c*) Determine iron, using one of the approved methods referred to in the note under Section 1.

Titania

12. Determine TiO_2 colorimetrically in the 25-ml. portion reserved for this purpose (Section 11) by oxidizing both the sample and the standard TiO_2 solution with several drops of a H_2O_2 solution (30 per cent). Compare the colors either in Nessler tubes or in a suitable colorimeter. Use a H_2SO_4 solution (5 per cent) for diluting purposes in matching the colors.

Alumina

13. Subtract the calculated weight of Fe_2O_3 (Section 11 (*a*) or (*b*)), TiO_2 (Section 12), and SiO_2 (Section 9) from the weight of R_2O_3 (Section 10). The remainder is the weight of Al_2O_3 plus small amounts of the oxides which may include those previously mentioned in Section 10. These are generally considered as Al_2O_3 in reporting the analysis of fireclay refractories.

Lime

14. Evaporate the combined filtrates reserved (Section 10) for the determination of CaO and MgO to about 200 ml., add 10 to 15 ml. of the saturated ammonium oxalate solution and 2 to 3 ml. of NH_4OH. Heat for 1 to 2 hr., by which time the volume should be about 75 to 100 ml. Allow the precipitated calcium oxalate to settle. Decant through a dense filter paper (Whatman No. 40 or equivalent), taking care to retain the precipitate in the beaker, wash several times with warm water by decantation, and then wash the paper until free from soluble salts. Reserve the filtrate for the MgO determination (Section 15). Return the paper to the beaker containing the precipitate, add 100 ml. of a H_2SO_4 solution (5 per cent), warm, and titrate to a faint pink end point with standard 0.04 *N* $KMnO_4$ solution. A blank should be previously determined for the effect of the paper.

NOTE.—For greater accuracy a double precipitation should be made, in which case, after precipitating the calcium oxalate as described above, decant the liquid and wash the beaker and paper several times with warm water. Dissolve the precipitate on the paper with warm

HCl (1:4), allowing it to run into the beaker containing the major portion of the calcium oxalate. Wash the paper with hot water. To the solution (about 75 to 100 ml. in volume) add several milliliters of saturated $(NH_4)_2C_2O_4$ solution and NH_4OH in slight excess. Heat for 2 hr., filter, wash, and titrate as described above.

Magnesia

15. Evaporate the filtrate from the CaO determination (Section 14) to about 150 to 200 ml. and add 2 to 3 g. of diammonium phosphate ($(NH_4)_2HPO_4$), stir until dissolved, and then add NH_4OH until alkaline and then 20 ml. in excess. Allow the solution to stand overnight. Filter and wash with NH_4OH (5 per cent). Dissolve the precipitate on the paper with hot HCl (1:4), allowing it to run into the beaker containing the precipitate. Wash the paper with hot water. To the solution, which should be not more than 100 ml. in volume, add 0.1 to 0.2 g. of $(NH_4)_2HPO_4$. Make ammoniacal, and then add a slight excess while stirring constantly until the precipitate is well formed. Then add 10 ml. more of NH_4OH and allow to stand overnight or at least for 4 hr. Filter through a No. 40 Whatman paper or equivalent. Transfer the precipitate to the paper and wash well with NH_4OH (5 per cent). Place the paper in a weighed platinum crucible, burn off the paper at a low temperature (below 900 C.), and ignite to constant weight at 1050 to 1100 C. (15 to 30 min. is sufficient).

Alkalies[7]

16. (*a*) Weigh 1.00 g. of the moisture-free (105 to 110 C.) sample (ground to an impalpable powder) and 3.0 g. of NH_4Cl into an agate mortar and mix well. Add 7 to 8 g. of $CaCO_3$ and again mix intimately. Place a $\frac{1}{8}$-in. layer of $CaCO_3$ in the bottom of a platinum crucible, and then add the above mixture, tapping the crucible occasionally to obtain a dense mass. Place a $\frac{1}{8}$-in. layer of $CaCO_3$ on the top. Heat the crucible over a low flame until ammonia fumes are no longer given off, then increase the heat so that the bottom half of the crucible is a dull red and maintain this temperature for about 1 hr. Cool, fill the crucible three-fourths full of water, and heat until the contents can be taken out and crushed in an agate mortar. Transfer to a platinum or porcelain dish by means of a jet of water. Evaporate to a low volume, decant through a No. 40 Whatman filter paper, or equivalent, and wash the material in the dish several times by decantation with warm water. Transfer to the paper and wash several times with hot water. Acidify with several milliliters of HCl and evaporate to a volume of 150 to 200 ml. Add several milliliters of NH_4OH and sufficient $(NH_4)_2CO_3$ to precipitate the lime, keeping the dish covered with a watch glass. Warm until the precipitate settles out. Filter and wash with warm water. Evaporate the solution to a low volume, then add a small lump of $(NH_4)_2CO_3$ to determine whether practically all calcium has been precipitated. If no precipitate forms, evaporate to dryness, otherwise precipitate and filter as before. Drive off the ammonium salts by heating just short of a dull red. Dissolve the residue in water and add a few milliliters of a saturated solution of $(NH_4)_2C_2O_4$ and 1 to 2 ml. of NH_4OH to precipitate the last trace of calcium. Heat for 30 to 45 min., filter, and wash with water containing $(NH_4)_2C_2O_4$ (0.1 per cent). Catch the filtrate in a weighed platinum dish. Add several drops of HCl and evaporate to dryness. Ignite gently as before and weigh as NaCl and KCl.

(*b*) The separation of the potassium and sodium must be carried out in an atmosphere free from ammonia fumes. Add to the solution of the combined chlorides in a small porcelain dish, sufficient chloroplatinic acid solution to re-

[7] This procedure for the determination of alkali is the J. Lawrence Smith method.

act with all of the sodium and potassium. The necessary amount to use is readily calculated from the known strength of the platinum solution and the weight of mixed chlorides counted as NaCl. The dilution of the resulting solution should be such that when heated on the steam bath any precipitate that may have formed entirely dissolves. This is to prevent inclusion of mother liquor in a mass of crystals suddenly formed. Evaporate until the solution is just syrupy enough to solidify on cooling. Do not evaporate to dryness, as this will dehydrate the sodium salt and render it less soluble in alcohol. Drench the residue with alcohol (80 per cent), filter by decantation through a small paper, wash by decantation with more of the alcohol, crushing the crystals with a small pestle or a widened and rounded end of a short glass rod. Reserve the filtrate and washings if sodium is to be determined directly. The residue should be golden yellow. An orange-red color indicates incomplete removal of the sodium salt. It is unnecessary to bring the mass of the precipitate upon the filter. Dry the dish and paper for a few moments to remove adhering alcohol. Dissolve the precipitate on the filter with hot water, catching the solution in a weighed crucible or small dish of platinum. Evaporate to dryness and add the salt that is still in the porcelain dish. If the salt is at all lumpy, redissolve it in water and again evaporate to dryness. Heat for 1 hr. at 130 C. in an air oven (100 C. suffices for very small amounts of fine-grained precipitate). It is necessary to cover the receptacle at first because the precipitate is prone to decrepitate. When dry, cool and weigh as K_2PtCl_6. Calculate the oxides, as follows:

$$KCl = \text{wt. of } K_2PtCl_6 \times 0.3068$$
$$K_2O = \text{wt. of } K_2PtCl_6 \times 0.1941$$
$$NaCl = \text{total chlorides} - KCl$$
$$Na_2O = NaCl \times 0.5303$$

SILICA REFRACTORIES

Special Solutions and Reagents Required

17. (*a*) *Sulfuric Acid* (*25 per cent by volume*).

(*b*) *Nitric Acid* (*50 per cent by volume*).

(*c*) *Standard Potassium Permanganate* (*0.01 N*).—Dissolve 0.325 g. in 1000 ml. of water. Standardize against the National Bureau of Standards' standard sample 40*c* of sodium oxalate.

Moisture

18. Weigh 1.00 g. of the sample and heat to constant weight at a temperature not under 105 nor over 110 C. Record the loss in weight as moisture.

Loss on Ignition

19. Weigh 1.000 g. of the moisture-free (105 to 110 C.) sample and heat to constant weight over a blast lamp, or in an electric muffle furnace, at 900 to 1000 C. Record the loss in weight as the ignition loss.

Silica

20. Transfer 1.0000 g. of the moisture-free (105 to 110 C.) sample to a 30-ml. platinum crucible. Add 10 ml. of HNO_3 (1:1) and 5 ml. of HF. Cover the crucible, place on a hot plate on an asbestos pad, and heat. Heat gently at first, gradually increasing the temperature. The final temperature should be regulated to prevent boiling or spattering. Heating in this manner for 1 hr. is sufficient to decompose the sample. Rinse the underside of the crucible lid with a few drops of water, allowing it to run into the crucible. Evaporate the crucible contents to dryness. Cool, add 10 ml. of HNO_3 (1:1), and again evaporate to dryness. Introduce the acid slowly by pouring it down the sides of

the crucible. Evaporate three times with the HNO_3 to insure complete volatilization of the fluorides. Ignite gently at first and then finally to constant weight at about 1200 C. Cool in a desiccator, and weigh. The loss in weight minus the loss on ignition (Section 19) represents all but a possible trace of the SiO_2 in the sample.

Iron and Aluminum Oxides

21. Fuse the residue in the crucible from the silica determination (Section 20) by heating gently with about 2 g. of potassium pyrosulfate, $K_2S_2O_7$, or sodium pyrosulfate, $Na_2S_2O_7$; cool, dissolve in water, and acidify with 3 to 4 ml. of HCl. Dilute to 150 to 200 ml., add 5 g. of NH_4Cl and 3 drops of methyl red (0.1 per cent). Heat to boiling, add NH_4OH (1:1) slowly, finally drop by drop until the solution is just alkaline to the indicator. Boil for 1 to 2 min. and filter through a 9-cm. rapid filter paper. Do not wash. Reserve the filtrate. Transfer the paper and precipitate to the beaker in which the precipitation was made, add 50 ml. of HCl (1:4), and digest covered on the hot plate, shaking frequently until the precipitate is dissolved and the paper well macerated. Dilute, add methyl red, reprecipitate with NH_4OH (1:1), boil, and filter, exactly as before. Add this second filtrate to the first filtrate for the subsequent determination of lime (Section 25). Swab the beaker and rod, and wash the paper and precipitate with NH_4Cl (2 per cent). Dry, then ignite the paper and precipitate to constant weight in a tared platinum crucible of 30-ml. capacity at 1000 to 1100 C. with access to air. Cool in a desiccator, and weigh as R_2O_3 which is mainly the iron and aluminum oxides. To recover the trace of SiO_2 which may have remained with the R_2O_3 group, add 2 drops of H_2SO_4 (1:4) and 1 ml. of HF to the crucible and evaporate to dryness. Cover the crucible and ignite to constant weight at about 1100 C. Cool and weigh. The loss in weight represents additional SiO_2 which should be added to that determined in Section 20.

Iron Oxide

22. Transfer 1.00 g. of the moisture-free (105 to 110 C.) sample to a 30-ml. platinum crucible and moisten with a few drops of water. Add 3 ml. of HNO_3, and about 15 ml. of HF. Evaporate at a moderate heat to dryness, taking care not to allow any loss by spattering. Decompose the nitrates and fluorides by ignition at about 1000 C. for 15 min. Now add to the residue in the crucible a small piece of $K_2S_2O_7$ or $Na_2S_2O_7$, and fuse. Dissolve the fusion in 25 ml. of HCl (1:1) and heat to boiling. Reduce the iron by adding $SnCl_2$ solution drop by drop from a pipette with constant swirling of the beaker until the solution is colorless, then add 1 drop in excess. Cool quickly in running water, then add at one stroke 15 ml. of saturated $HgCl_2$ solution. Allow to stand for 3 min., transfer with washing to a 1000-ml. beaker containing 300 ml. of cold distilled water and 25 ml. of $MnSO_4$ solution. Titrate with standard 0.01 *N* $KMnO_4$, added very slowly while stirring constantly, until a permanent pink end point is obtained.

Titania

23. Fuse the ignited R_2O_3 residue after recovery of SiO_2 (Section 21) with a very small piece of $K_2S_2O_7$ or $Na_2S_2O_7$. Cool, and dissolve the fusion by the addition of about 25 ml. of warm water and 1 to 2 ml. of H_2SO_4 to the crucible. It is important at this point to keep the solution to a small volume. Transfer to a 50-ml. volumetric flask, add a few drops of H_2O_2, and dilute to the mark. Place the 50 ml. of solution in a small

Nessler tube. Compare the color of this solution with the color of a known standard solution. A satisfactory solution is one of such strength that 1 ml. equals 0.0001 g. of TiO_2. To make the comparison use any standard colorimeter, or place an appropriate amount of standard solution in a second Nessler tube and dilute with water from a burette until the color is matched. From the amount of water added, calculate the percentage of TiO_2 in the sample.

Alumina

24. Subtract the calculated weight of Fe_2O_3 (Section 22), the weight of SiO_2 recovered (Section 21), and the calculated weight of TiO_2 (Section 23) from the weight of R_2O_3 (Section 21). The remainder is the weight of the Al_2O_3, plus small amounts of oxides of any other elements that were precipitated.

NOTE.—These may include P_2O_5 and ZrO_2, but they are present in such small amounts as to be disregarded in the complete analysis of silica refractories.

Lime

25. Acidify the combined filtrate from the R_2O_3 precipitation with several milliliters of HCl (1:1) (Section 21) and evaporate to a volume of about 250 ml. Heat to incipient boiling, add 25 ml. of a saturated $(NH_4)_2C_2O_4$ solution and NH_4OH until slightly alkaline. Stir thoroughly and allow the precipitate to settle out, keeping the solution warm for 3 to 4 hr. but not boiling. Filter and wash five times with cold water. Save the filtrate. Dissolved the washed oxalate precipitate with 50 ml. of diluted HCl (1:1), dilute to about 150 ml., and precipitate as before. Wash the beaker, precipitate, and paper with cold water until $(NH_4)_2C_2O_4$ is removed. Avoid excessive washing. Combine the filtrates for the magnesia determination (Section 26). To the beaker used for the precipitation, add 150 ml. of a H_2SO_4 solution (5 per cent), then introduce the paper containing the precipitate. Heat to almost boiling and titrate with 0.1 *N* $KMnO_4$. A blank should be previously determined for the effect of the paper.

Magnesia

26. To the combined filtrates from the lime determination (Section 25), which have been reduced by evaporation to about 300 ml., add 2 to 3 g. of $(NH_4)_2HPO_4$. Stir until dissolved and then add NH_4OH slowly until alkaline, and then 30 ml. of NH_4OH in excess. Allow the solution to stand for at least 4 hr., and preferably overnight if the total amount of solution exceeds 400 ml. in volume. Filter, wash with diluted NH_4OH (5 per cent). Dissolve from the paper with hot HCl (1:1) and reprecipitate by adding 0.1 g. of $(NH_4)_2HPO_4$, diluting, rendering ammoniacal, and letting stand as before. After filtering and washing as before, char the paper and burn off the carbon below 900 C., and then ignite in an electric muffle furnace at about 1100 C. to constant weight. The ignited material consists of magnesium pyrophosphate, $Mg_2P_2O_7$. Calculate the weight of MgO by multiplying the $Mg_2P_2O_7$ by 0.3621.

Alkalies

27. Determine the alkalies by the procedure described in Section 16 (*a*) and (*b*) for the analysis of fireclay refractories.

HIGH-ALUMINA REFRACTORIES

(Includes Bonded Refractories made from Diaspore, Fused Alumina, and the Sillimanite Minerals, as well as Electrocast.)

Special Solutions Required

28. (*a*) *Potassium Nitrate (1 per cent).*—Dissolve 1 g. in 100 ml. of water.

(*b*) *Molybdate Reagent.*—Mix 100 g. of pure molybdic anhydride or 118 g. of molybdic acid (85 per cent) with 400 ml. of water, add 80 ml. of NH_4OH, and filter when solution is complete. Prepare a second solution containing 400 ml. of HNO_3 and 600 ml. of water. Agitate the HNO_3 solution by means of a current of air and add the molybdate solution very slowly. Continue agitation for 1 hr. Add a small crystal of $(NH_4)_2HPO_4$, let stand over night, filter, and keep in a glass-stoppered bottle.

(*c*) *Standard Sodium Hydroxide Solution.*[4a]—Dissolve 60 g. of NaOH in 100 ml. of distilled water in a 150-ml. test tube of resistant glass. Stopper tightly with a cork covered with tin foil and let stand until the supernatant liquid is clear. By means of a pipette take out 5.0 ml. of the clear solution and dilute to 1 liter with freshly boiled water (Note).

(*d*) *Standard Nitric Acid Solution.*[4a]—Dilute 5.0 ml. of HNO_3 (sp. gr. 1.42) to 1 liter with freshly boiled water (Note).

NOTE.—Adjust the NaOH and HNO_3 solutions until they exactly agree, and standardize against National Bureau of Standards' standard sample No. 78 of known phosphorous content.

Moisture

29. Weigh 1.00 g. of the sample and heat to constant weight at a temperature not under 105 nor over 110 C. Record the loss in weight as moisture.

Loss on Ignition

30. Weigh 1.000 g. of the moisture-free (105 to 110 C.) sample and heat to constant weight over a blast lamp, or in an electric muffle furnace, at 900 to 1000 C. Record the loss in weight as the ignition loss.

Silica

31. (*a*) Weigh 1.0000 g. of the moisture-free (105 to 110 C.) sample into a platinum crucible which is free from any iron stain. Add 0.40 g. of anhydrous borax ($Na_2B_4O_7$) and 2.00 g. of Na_2CO_3. Mix well and fuse first at a low red heat, gradually increasing to the full temperature of a blast burner. Blast for 15 min. Dissolve the melt in diluted HCl (1:4) in an evaporating dish, add 2 to 3 ml. of diluted H_2SO_4 (1:1) (Note 1), and evaporate to dryness. Wash down the sides of the dish with 25 ml. of methyl alcohol (Note 2) saturated with HCl gas. Evaporate, add methyl alcohol solution, and evaporate again. Bake to dehydrate the SiO_2. Cool, add 15 ml. of HCl and 50 ml. of water, boil 1 to 2 min., and filter on a No. 40 Whatman or similar filter paper. Wash four to five times with hot diluted HCl (1:4), then seven to eight times with hot water. Reserve the paper containing the SiO_2 precipitate. Transfer the filtrate to the original dish and evaporate to dryness and bake. Add 15 ml. of HCl and 50 ml. of water, bring just to a boil, filter on a No. 42 Whatman or similar filter paper, and wash as before. Reserve the filtrate.

NOTE 1.—Diluted H_2SO_4 is used to prevent TiO_2 from hydrolyzing and precipitating with the SiO_2.

NOTE 2.—Methyl alcohol is used to eliminate traces of boron.

(*b*) Place the two filter papers containing the SiO_2 precipitate in a platinum crucible, ignite carefully until the paper is burned off, then heat at 1100 to 1200 C. for 10 to 15 min. Cool, weigh, and repeat heating at 1100 to 1200 C. until constant weight is obtained. Moisten the ignited precipitate with water, add HF slowly until the crucible is half-full, then add 5 drops of H_2SO_4 and evaporate to fumes. Cool, add 2 to 3 ml. of HF and 2 drops of H_2SO_4, and evaporate to dryness. Heat the crucible to bright redness and blast at 1100 to 1200 C. for 5 min. Cool, weigh, and repeat blasting until constant weight is

obtained. The loss in weight represents the SiO_2 content except for the small amount which is later recovered from the alumina precipitate.

Separation of Al_2O_3 and P_2O_5 from Fe_2O_3, TiO_2, CaO, and MgO

32. Fuse the residue from the HF evaporation (Section 31 (*b*)) with a little $K_2S_2O_7$ or $Na_2S_2O_7$, dissolve the melt in diluted HCl (1:4) and add to the filtrate from the SiO_2 precipitate which was reserved. Reduce the volume to about 75 ml. and nearly neutralize with NaOH (10 per cent). Slowly and while swirling constantly pour into a 250-ml. volumetric flask containing 100 ml. of NaOH (10 per cent) to which has been added 1 g. each of Na_2CO_3 and Na_2O_2 (Notes 1 and 2). Warm on a water bath for 1 hr. and cool to room temperature. Dilute with water to the mark, mix well, and let stand a few minutes. Filter off the precipitate on a filter paper which has been previously washed with NaOH (10 per cent) and dried, catching the filtrate in a dry beaker. Do not wash the precipitate. From the filtrate take a 100-ml. aliquot for the determination of Al_2O_3 (Section 36) and another 100-ml. aliquot for the determination of P_2O_5 (Section 35).

NOTE 1.—The Na_2CO_3 is used to precipitate calcium and magnesium along with iron and titanium.

NOTE 2.—The Na_2O_2 is used to oxidize any chromium present so that it may later be separated from the Al_2O_3.

Iron Oxide

33. Dissolve the precipitate on the filter paper and that adhering to the flask with hot diluted HCl (1:4). Adjust the volume to about 100 ml., add 5 g. of NH_4Cl, and heat to boiling. Add NH_4OH while stirring slowly until there is a slight excess. Again heat to boiling, filter hot, and wash five times with hot NH_4Cl (2 per cent). Place the paper containing the precipitate in the original beaker, add 15 ml. of HCl, stir to macerate the paper, dilute with hot water to 100-ml. volume, and precipitate with NH_4OH. Filter, and wash as before. Combine the two filtrates and reserve for the determination of CaO and MgO. Dissolve the precipitate in hot diluted HCl (1:4), and cool. Pour into a 100-ml. volumetric flask, dilute with water to the mark, and mix. Draw off exactly 5.0 ml. of the solution and reserve for the determination of TiO_2 (Section 34). Pour the remaining solution into a 250-ml. beaker and evaporate to dryness. Add 25 ml. of HCl (1:1), and heat to boiling. From this point proceed in accordance with the procedure described in Section 22 for the determination of Fe_2O_3 in the analysis of silica refractories.

Titania

34. Add to the 5.0-ml. portion previously reserved (Section 33) 10 ml. of H_2SO_4 (1:1) and evaporate to fumes. Dilute with 10 to 20 ml. of water, and cool. Pour into a 100-ml. volumetric flask, add a few drops of H_2O_2 (30 per cent), dilute to the mark, and mix. To a 100-ml. volumetric flask, add exactly 20.0 ml. of the standard TiO_2 solution and 10 ml. of H_2SO_4 (1:1). Add a few drops of H_2O_2 (30 per cent), dilute to the mark, and mix. Compare the colors either in Nessler tubes or in a suitable colorimeter.

Phosphorous Oxide

35. Acidify with HNO_3 the 100-ml. aliquot previously reserved (Section 32). Dilute to 200-ml. volume and precipitate the Al_2O_3 and P_2O_5 by adding a slight excess of NH_4OH solution. Filter, wash a few times with hot water, and dissolve the precipitate in 100 ml. of diluted HNO_3 (1:9). Transfer to a 300-ml. Erlenmeyer flask, heat to boiling and add a saturated solution of $KMnO_4$

until a pink color persists. Decolorize with a few drops of sulfurous acid, add 15 ml. of HNO_3, cool, adjust the volume to about 125 ml., and add 20 ml. of NH_4OH and 40 ml. of molybdate reagent. Shake for 10 min., let stand for 30 to 40 min., and filter. Wash the flask, paper, and precipitate at least ten times with KNO_3 solution (1 per cent). Return the paper and precipitate to the flask. Add an excess of standard NaOH solution and 25 ml. of water (both free from CO_2). Shake until the precipitate is dissolved and dilute to a 100-ml. volume. Add 2 to 3 drops of phenolphthalein (0.2 per cent), then titrate with standard HNO_3 to the disappearance of the pink color. Subtract from the original NaOH added the volume equivalent to the HNO_3 required to discharge the color. The difference gives the milliliters of NaOH equivalent to the P_2O_5 present. Calculate the percentage of P_2O_5.

Alumina[5a]

36. (*a*) Dilute the 100-ml. aliquot, previously reserved (Section 32), to a 250-ml. volume, make acid with HCl (1:1), and add 5 g. of NH_4Cl. Heat to boiling, add NH_4OH very slowly, while stirring constantly, until there is a slight excess as shown by methyl red. Boil 1 to 2 min., filter hot, and wash five to six times with hot NH_4Cl (2 per cent). Place the paper containing the precipitate in the original beaker, add 15 ml. of HCl, and, with the aid of a glass stirring rod, macerate the paper. Add boiling water to make up to a volume of about 250 ml. Repeat the precipitation with NH_4OH and filter as before. Wash thoroughly with hot NH_4Cl (2 per cent), using at least 250 ml. of wash solution.

(*b*) Place the paper containing the precipitate in a platinum crucible which has been previously weighed with its cover. Carefully burn off the paper, blast at not less than 1200 C. for 10 min., cool, and weigh. Repeat blasting and weighing until constant weight is obtained. The increase in weight is the Al_2O_3 and P_2O_5 plus a little SiO_2 which has escaped precipitation. To obtain the percentage of Al_2O_3, subtract the P_2O_5 (Section 35) and SiO_2 (Section 36 (*c*)).

(*c*) To determine the SiO_2, fuse the precipitate with about 5 g. of $K_2S_2O_7$ or $Na_2S_2O_7$. Dissolve the melt in warm water, add about 30 ml. of diluted H_2SO_4 (1:1) solution. Evaporate to copious fumes of H_2SO_4 and cool. Take up with water, filter, wash, ignite in a platinum crucible, and weigh. Treat the residue with 5 ml. of HF and 2 to 3 drops of H_2SO_4, evaporate to dryness, ignite, and weigh. The loss in weight is SiO_2. Add to the original silica determination.

Lime

37. Determine CaO in the filtrate from the Fe_2O_3 precipitate according to the method described for the determination of lime under analysis of fireclay refractories (Section 14).

Magnesia

38. Determine MgO by the method described for the determination of magnesia under analysis of fireclay refractories (Section 15).

Alkalies

39. Proceed as in the determination of alkalies under the analysis of fireclay refractories (Section 16) but with the following precautions:

(*a*) Fuse the sample at 1200 C. to obtain complete decomposition of the material.

(*b*) Cool the top of the crucible to prevent any loss of alkalies by volatilization. Cooling can be accomplished more

easily if a platinum crucible of the J. Lawrence Smith, long type is used.

MAGNESITE REFRACTORIES

Special Solutions Required

40. The following solutions will be required:

(*a*) *Hydrochloric Acid (2.5 per cent by volume).*

(*b*) *Potassium Permanganate (Approximately 0.5 N).*—Dissolve 16 g. of $KMnO_4$ in 1000 ml. of water.

(*c*) *Ammonium Chloride - Hydrochloric Acid Solution.*—Dissolve 50 g. of NH_4Cl in water, add 50 ml. of HCl (sp. gr. 1.19), and dilute to 1000 ml.

Preparation of Samples for Analysis

41. A 10-lb. sample carefully obtained after the manner of one of the approved methods of sampling shall be crushed to pass a No. 8 (2380-micron) sieve,[5] mixed thoroughly, and reduced by quartering to about 0.5 lb. This shall be crushed to pass a No. 20 (840-micron) sieve,[5] mixed, and quartered to about 50 g. This 50-g. sample shall then be ground in an agate mortar so that it will all pass a No. 100 (149-micron) sieve,[5] mixed thoroughly, and placed in an airtight container until analyzed, since finely ground, dead-burned magnesite gradually absorbs and fixes moisture and carbon dioxide.

Moisture

42. Weigh 1.00 g. of the sample and heat to constant weight at a temperature not under 105 nor over 110 C. Record the loss in weight as moisture.

Loss on Ignition

43. Weigh 1.000 g. of the moisture-free (105 to 110 C.) sample and heat to constant weight over a blast lamp, or in an electric muffle furnace, at 900 to 1000 C. Record the loss in weight as the ignition loss.

Silica

44. Transfer 1.0000 g. of the moisture-free (105 to 110 C.) sample to a 210-ml. casserole. Moisten the solids with a few milliliters of water, slowly add 20 ml. of HCl, and cover the casserole. While covered, digest on the hot plate until solution is complete and all gritty particles have disappeared, breaking up any solid cake from time to time with a stirring rod. Uncover, add 8 ml. of perchloric acid, $HClO_4$ (60 to 70 per cent), and evaporate to heavy white fumes of $HClO_4$. Cover and boil so that the acid refluxes for 10 to 15 min. Remove from the hot plate, cool, wet with a few milliliters of water and 5 ml. of HCl, warm for a few minutes on the hot plate, add 50 ml. of hot water, heat to approximately boiling, and filter through a 9-cm. rapid filter. Wash the paper and precipitate ten times with a hot HCl solution (2.5 per cent), then three times with hot water. Return the filtrate to the casserole in which the first evaporation was made, add 2 ml. of $HClO_4$, and evaporate to fumes on the hot plate, boiling as before. Cool, take up in acid and water, filter, and wash as before. Reserve the filtrate. Place both papers and precipitates in a tared platinum crucible. Dry, then burn off the papers and ignite to constant weight in the muffle or over a moderate blast. Cool in a desiccator and weigh as impure SiO_2. Add two drops of diluted H_2SO_4 (1:1) and 1 ml. of HF to the impure SiO_2 in the crucible. Digest for 5 to 10 min., covered, on the hot plate, then remove the cover and evaporate to dryness. Fume off the H_2SO_4 over a moderate heat, and ignite or blast for a few minutes. Cool in a desiccator, and weigh again. Subtract the weight of the

crucible and residue from the weight of the crucible and impure SiO_2; the difference is the corrected SiO_2. Fuse the residue in the crucible by heating gently with a small piece of $K_2S_2O_7$ or $Na_2S_2O_7$ about the size of a pea, cool, dissolve in water, and add to the reserved filtrate from the silica determination.

Iron and Aluminum Oxides

45. Add to the filtrate from the silica determination (Section 44), which should be in a 400-ml. beaker, 5 g. of NH_4Cl. Dilute from 150 to 200 ml., add three drops of methyl red (0.1 per cent), heat to boiling. Add NH_4OH (1:1) slowly, finally drop by drop until the solution is just alkaline to the indicator. Boil for 1 or 2 min. and filter through a 9-cm. rapid filter. Do not wash. Reserve the filtrate. Transfer the paper and precipitate to the beaker in which the precipitation was made, add 50 ml. of HCl (1:4), and digest covered on the hot plate, shaking frequently until the precipitate is dissolved and the paper well macerated. Dilute, add methyl red, reprecipitate with NH_4OH (1:1), boil, and filter, exactly as before. Add this second filtrate to the first filtrate for the subsequent determination of lime (Section 48 (*a*)). Swab the beaker and rod and wash the paper and precipitate with NH_4Cl solution (2 per cent). Dry, then ignite the paper and precipitate in a tared platinum crucible at 1000 to 1100 C. for 1 hr. with access of air. Cool in a desiccator, and weigh as R_2O_3 which is mainly the Fe_2O_3 and Al_2O_3.

Iron Oxides

46. Weigh 2.00 g. of the moisture-free (105 to 110 C.) sample into a 150-ml. beaker, and cover. Dissolve by slowly adding HCl, keeping the beaker on the hot plate. Finally, add about 5 ml. of HCl in excess. Heat to gentle boiling until the siliceous residue undissolved appears iron-free. Filter, and fuse the residue with a very little $K_2S_2O_7$ or $Na_2S_2O_7$ in a porcelain or vitreous silica crucible to avoid introducing platinum. Dissolve the fused material and add to the filtrate. Add a few drops of a strong solution of $KMnO_4$ (about 0.5 *N*) to oxidize any organic matter that may be present. Boil. When the excess of $KMnO_4$ is decomposed and chlorine fumes have disappeared, reduce the iron by adding $SnCl_2$ solution drop by drop from a pipette while swirling the beaker constantly until the solution is colorless, then add 1 drop in excess. Cool quickly in running water, then add at one stroke 15 ml. of saturated $HgCl_2$ solution. Allow to stand for 3 min., transfer with washing to a 600-ml. beaker containing 300 ml. of cold distilled water and 25 ml. of $MnSO_4$ solution. Titrate with standard 0.1 *N* $KMnO_4$ solution, added very slowly while stirring constantly until a permanent pink end point is obtained.

Alumina

47. Subtract the calculated weight of iron oxide (Section 46) from the weight of R_2O_3 (Section 45). The remainder is the weight of the Al_2O_3 plus small amounts of oxides of any other elements that were precipitated (Note).

NOTE.—These may include P_2O_5, TiO_2 and small amounts of SiO_2, but they are present in such small amounts as to be disregarded in the complete analysis of magnesite refractories.

Lime

48. (*a*) *Method A: When MgO is to be Determined Directly.*—Acidify with several milliliters of HCl (50 per cent) the combined filtrate from the R_2O_3 precipitation (Section 45) and evaporate to a volume of about 250 ml. Heat to incipient boiling, add 25 ml. of a saturated $(NH_4)_2C_2O_4$ solution (about 4 per cent) and NH_4OH until slightly alkaline. Stir thoroughly and allow the precipitate

to settle out, keeping the solution warm for 3 to 4 hr. but not boiling. Filter and wash five times with cold water. Save the filtrate. Dissolve the washed oxalate precipitate with 50 ml. of diluted HCl (1:1), dilute to 250 ml. and precipitate as before. Wash the beaker, precipitate, and paper with cold water until $(NH_4)_2C_2O_4$ is removed. Avoid excessive washing. Combine the filtrates for the determination of MgO (Section 49). To the beaker used for the precipitation, add 150 ml. of H_2SO_4 (5 per cent), then introduce the paper containing the precipitate. Heat to almost boiling and titrate with 0.1 *N* $KMnO_4$. A blank should be previously determined for the effect of the paper.

A small part of the CaO occasionally escapes the oxalate precipitation and comes down subsequently with the MgO. To the CaO precipitated by the oxalate should accordingly be added that found in the MgO.

(*b*) *Method B: When MgO is to be Determined by Difference.*[8]—Weigh 2.50 g. of the moisture-free (105 to 110 C.) sample into a 250-ml. beaker, add 10 ml. of water, cover, slowly add HCl until dissolved, stirring frequently and avoiding excessive boiling. Then add about 5 ml. of HCl in excess. Evaporate on a hot plate until very thick and syrupy. At this stage crusts will be forming on the surface and a drop of material placed on the wall of the beaker will immediately solidify. Remove the beaker from the hot plate and allow to partially cool. Add 5 ml. of H_2SO_4 (10 per cent). Cover with a watch glass, heat on a hot plate until all soluble matter is dissolved. Cool slightly, but not until solids separate. Add 50 ml. of alcohol, stir thoroughly, and heat just to boiling on a hot plate. Filter with suction through a Gooch crucible prepared with a moderately thin asbestos mat. If the SiO_2 is less than 3.5 per cent, filter through paper. Rinse the beaker twice with alcohol, pouring the washings through the crucible. Drain under suction. Remove the crucible, scrape out the mat with a glass rod into the beaker in which the precipitation of sulfate was made, wash the crucible clean with a hot NH_4Cl - HCl solution, using about 35 ml. If the SiO_2 is high (over 6 per cent) it is advisable to filter it off at this point before precipitating the lime. Dilute to about 150 ml. with hot water, make alkaline with NH_4OH, heat to boiling, and precipitate the calcium by slowly adding 15 ml. of a saturated solution of $(NH_4)_2C_2O_4$. Digest on the hot plate just below boiling for 30 min. Filter on a 9-cm. paper, wash with cold water until free from oxalate, testing final washing with a drop of $KMnO_4$ after acidification. Transfer the paper and precipitate to the original beaker, dissolve with 150 ml. of H_2SO_4 (5 per cent), and titrate as in Method A (Paragraph (*a*)). Correct for a blank on the filter paper.

Magnesia[5a]

49. (*a*) Acidify with several milliliters of HCl (50 per cent) the combined filtrates reserved during the determination of CaO (Section 48 (*a*)). Dilute to 500 ml. in a calibrated flask and take a 50 ml. ($\frac{1}{10}$) aliquot portion. Since a 1-g. sample was originally used, this will yield aliquot portions containing between 0.08 to 0.09 g. of MgO. Dilute to 250 ml. and add 2 to 3 g. of diammonium phosphate ($(NH_4)_2HPO_4$), stir until dissolved. Add NH_4OH slowly until alkaline and then 30 ml. of NH_4OH in excess. Allow the solution to stand for at least 4 hr., preferably overnight if the total amount of solution exceeds 400 ml. in

[8] In this specific procedure very much less lime escapes precipitation and thus the magnesia "by difference" is not subject to this plus error. The procedure further has the advantage that an individual lime determination may be made without separating the other usual constituents.

volume. Filter, wash with diluted NH_4OH (5 per cent). Dissolve from the paper with hot HCl (1:1) and reprecipitate by adding 0.1 g. of $(NH_4)_2HPO_4$, diluting, rendering ammoniacal, and letting stand as before. After filtering and washing as before, char the paper and burn off the carbon below 900 C., then ignite to constant weight in an electric muffle furnace at about 1100 C. The ignited material consists of magnesium pyrophosphate, $Mg_2P_2O_7$, as well as some of the CaO and MnO. Correction may be made for these as follows:

(*b*) Dissolve the ignited pyrophosphate in a little diluted H_2SO_4 and add enough absolute alcohol to make up 90 to 95 per cent of the final volume. Any calcium present will settle out as $CaSO_4$. Filter, wash with alcohol, and dissolve the precipitate in hot diluted HCl. Precipitate with oxalate and titrate with permanganate. Add this CaO to that previously found. Calculate the CaO to tricalcium phosphate, $Ca_3(PO_4)_2$, by multiplying by the factor 1.845 and deduct this amount from the weight of the $Mg_2P_2O_7$. The correction to the first calcium precipitate is ten times the weight of the CaO precipitated as sulfate.

(*c*) The manganese which contaminates the $Mg_2P_2O_7$ is determined in the alcoholic filtrate from the $CaSO_4$. Evaporate this filtrate until strong fumes are evolved and all organic matter destroyed. Cool, dissolve the residue in 25 ml. of HNO_3 (30 per cent) and determine the manganese by the bismuthate method, as described in Section 50, omitting the Na_2CO_3 fusion. Calculate any manganese found to $Mn_2P_2O_7$ by multiplying by the factor 2.585 and deduct this weight from the $Mg_2P_2O_7$.

Manganese as MnO

50. Mix intimately 1.0 to 5.0 g. of the moisture-free (105 to 110 C.) sample (depending upon the amount of manganese expected) with 0.5 to 2.5 g. of anhydrous Na_2CO_3 and ignite in a platinum crucible for 15 min. at about 1000 C. Dissolve the sintered melt in 50 ml. of HNO_3 (30 per cent), and if there is any evidence of brown hydrated MnO_2 add sulfurous acid drop by drop. Boil the solution, filter if necessary, cool to room temperature or lower, and add 2 to 3 g. of sodium bismuthate. Agitate the solution for 1 min. and filter through asbestos (Note). Wash with HNO_3 (3 per cent) until the residue is free from permanganic acid, as determined by the absence of the pink color in the washings. Titrate, at once, the permanganic acid formed, with standard sodium arsenite (1 ml. = 0.0002 g. of Mn). The end point is the disappearance of the pink color. The factor for converting Mn to MnO is 1.291.

NOTE.—A good grade of asbestos, acid washed, and specially prepared for Gooch crucibles, should be used.

CHROME ORE AND CHROME REFRACTORIES

(*Including Chrome-Magnesite Refractories*)

NOTE.—This method is not intended to cover electrically fused aluminous refractories containing chrome ore.

Reagents

51. (*a*) *Ammonium Hydroxide* (*2.5 per cent by volume*).

(*b*) *Ammonium Hydroxide* (*10 per cent by volume*).

(*c*) *Ammonium Hydroxide* (*25 per cent by volume*).

(*d*) *Hydrochloric Acid* (*5 per cent by volume*).

(*e*) *Hydrochloric Acid* (*10 per cent by volume*).

(*f*) *Phosphoric Acid*, H_3PO_4 (*sp. gr. 1.71*).

(*g*) *Ammonium Chloride Solution* (*1 per cent*).—Dissolve 1 g. of NH_4Cl in 100 ml. of water.

(*h*) *Ammonium Persulfate Solution.*—Dissolve 25 g. of $(NH_4)_2S_2O_8$ in 80 ml. of water.

(*i*) *Standard Ferrous Ammonium Sulfate Solution (0.2 N).*—Dissolve 78.4 g. of $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ in 1 liter of water (Note). Standardize against 0.2 *N* $KMnO_4$. This standardization must be made each day the solution is used, as it gradually weakens due to oxidation.

NOTE.—C.P. ferrous ammonium sulfate crystals may be used instead of the standard solution.

(*j*) *Standard Potassium Permanganate (0.2 N).*—Dissolve 6.5 g. of $KMnO_4$ in 1 liter of water. Standardize against the National Bureau of Standards' standard sample 40*c* of sodium oxalate.

(*k*) *Potassium Permanganate Solution (2.5 per cent).*—Dissolve 2.5 g. of $KMnO_4$ in 100 ml. of water.

(*l*) *Silver Nitrate Solution (0.5 per cent).*—Dissolve 0.5 g. of $AgNO_3$ in 100 ml. of water.

(*m*) *Sodium Chloride Solution (10 per cent).*—Dissolve 10 g. of NaCl in 100 ml. of water.

(*n*) *Ortho-Phenanthroline Indicator (Ferroin).*

Preparation of Samples for Analysis

52. The 50-g. sample referred to in Section 3 shall be ground so that it will all pass a No. 200 (74-micron) sieve.[5]

Moisture

53. Weigh 1.00 g. of the sample and heat to constant weight at a temperature not under 105 nor over 110 C. Record the loss in weight as moisture.

Loss on Ignition

54. Weigh 1.000 g. of the moisture-free (105 to 110 C.) sample and heat to constant weight over a blast lamp, or in an electric muffle furnace, at 900 to 1000 C. Record the loss in weight as the ignition loss.

Chromium Oxide

55. (*a*) Transfer 0.5000 g. of the moisture-free (105 to 110 C.) sample to a 30-ml. pure iron crucible (Note 1). Mix with about 8 g. of dry Na_2O_2, cover with an iron lid, and heat over a very low flame (Note 2). After the charge is melted, increase the temperature slowly to a medium red (650 to 750 C.), and continue heating for 3 to 5 min. Do not continue heating longer than this, because attack of the flux is quite rapid at red heat or above. After removing the crucible from the heat, rotate to spread the contents over the crucible walls, and cool. Place crucible and lid in a covered 600-ml. beaker, and add 250 ml. of cold water. After reaction has stopped and the melt is completely dissolved, remove the crucible and lid, and wash them thoroughly (Note 3).

NOTE 1.—Ordinary iron crucibles, which may contain significant amounts of chromium, should be avoided. Crucibles made of ingot iron are satisfactory.

NOTE 2.—If about 0.2 g. of very finely pulverized sugar charcoal is mixed with the Na_2O_2, only about 5 g. of the peroxide is required. Not more than 0.2 g. of sugar charcoal is recommended in order to avoid the possibility of explosion. After heating for about 30 sec., ignition of the sugar charcoal takes place, and the crucible will suddenly become a dull red on the outside. The total time required to complete the fusion is about 2 min. By thus shortening the time for fusion and contact with the flux, the life of the crucible is extended.

NOTE 3.—If sugar charcoal is used, it is advisable to add 1 to 2 g. of Na_2O_2 at this point.

(*b*) Boil for 10 min., or digest at 80 to 90 C. for 30 to 40 min., stirring occasionally to decompose the excess Na_2O_2. Cool, and add 60 ml. of H_2SO_4 (1:1) and 5 ml. of HNO_3. Boil until the iron scale from the crucible is dis-

solved. Add 20 to 25 ml. of $AgNO_3$ (0.5 per cent), 1 to 2 drops of $KMnO_4$ (2.5 per cent), and 10 to 15 ml. of $(NH_4)_2S_2O_8$ solution. Boil for 5 min. Add 20 ml. of NaCl (10 per cent) and boil for 5 to 10 min. after the permanganate or MnO_2 has completely disappeared. Cool to room temperature, and add 3 ml. of H_3PO_4.

(*c*) Now add a measured excess of ferrous ammonium sulfate, either as c.p. crystals or as a standard solution. If the crystals are used, add exactly 5 g. and stir until completely dissolved. If a solution is used, add from a burette the standard 0.2 *N* solution with 5 ml. in excess. The chromium will thus be reduced to the trivalent state and will change the color of the solution from amber to a bright green. Add two drops of ortho-phenanthroline (Ferroin) as an indicator. (The solution will become turbid and pink in color.) Titrate the ferrous ammonium sulfate, in excess of that required to reduce the chromium, with 0.2 *N* $KMnO_4$. The end point is reached when one drop of the standard $KMnO_4$ solution causes the turbidity to disappear and the solution to become clear green in color. Designate the first titration in either case as T_1. If ferrous ammonium sulfate crystals were used for reduction, titrate 5 g. of the crystals dissolved in H_2SO_4 (5 per cent) with the 0.2 *N* $KMnO_4$ solution. Designate the second titration as T_2. If the standard ferrous ammonium sulfate solution was used, T_2 is the milliliters of 0.2 *N* $KMnO_4$ equivalent to the total volume of 0.2 *N* ferrous ammonium sulfate originally added, including the 5 ml. excess. In either case, $T_2 - T_1$ then equals the milliliters of 0.2 *N* $KMnO_4$ equivalent to the weight of ferrous ammonium sulfate required to reduce the chromium in the sample. Calculate the percentage of chromium oxide, as follows:

$$Cr_2O_3, \text{ per cent} = \frac{(T_2 - T_1) \times N \text{ of } KMnO_4 \times 0.02534}{W} \times 100$$

where:

0.02534 = Cr_2O_3 equivalent of the $KMnO_4$, and

W = weight of the sample in grams.

Titania and Ferrous Oxide

56. (*a*) Transfer 0.5000 g. of the moisture-free (105 to 110 C.) sample to a 30-ml. nickel crucible. Mix with 2 to 3 g. (Note) of dry Na_2O_2, cover with lid, and fuse as described in Section 55.

NOTE.—The use of only 2 to 3 g. of Na_2O_2 reduces the amount of nickel dissolved which might interfere with the subsequent colorimetric determination of TiO_2. The small amount of nickel actually dissolved is entirely removed by the double NH_4OH precipitation.

(*b*) Take up the fusion in about 250 ml. of cold water. After reaction has stopped and the soluble constituents of the melt have completely dissolved, remove the crucible and cover and wash thoroughly. Boil for 5 min. Filter the iron and titanium oxides, and wash 10 times with hot water. Dissolve the precipitate from the paper with diluted HCl (1:1), and precipitate with NH_4OH in excess. Filter, and wash thoroughly with hot water. Again dissolve with HCl (1:1) and reprecipitate with excess NH_4OH, filter, and wash. Finally, dissolve the precipitate with hot diluted H_2SO_4 (10 per cent), cool, add several drops of H_2O_2, and dilute to 250 ml.

Titania

NOTE.—This determination is optional, since it is made only infrequently.

57. Take a 100-ml. aliquot portion from the 250-ml. acid solution (Section 56) and determine the TiO_2 colorimetrically by placing the solution in a 100-ml. Nessler tube (Note) and comparing it with the color of an appropriate

amount of the standard TiO_2 solution in another Nessler tube. Both the solution of the sample and the reference standard solution shall be cooled to room temperature before making the comparison. Dilute the reference standard with a measured amount of water from a burette until the color is matched. From the amount of water added, calculate the percentage of TiO_2 in the sample.

NOTE.—If preferred, any standard colorimeter may be used instead of the Nessler tubes.

Ferrous Oxide

58. (*a*) Use the remaining 150-ml. portion of the acid solution (Section 56), but boil off the H_2O_2; or, if TiO_2 is not to be determined, use the entire 250-ml. of solution (without adding H_2O_2).

NOTE.—It is equally satisfactory to add the 100-ml. aliquot portion used for the TiO_2 determination in Section 57 to the remaining 150 ml. in order to obtain the advantage in accuracy of using the entire 0.5-g. original sample.

(*b*) Evaporate to 100 ml. or less, add 25 ml. of diluted HCl (1:1), and heat to boiling. Reduce the iron by adding $SnCl_2$ solution drop by drop from a pipette, while swirling the beaker constantly, until the solution is colorless. Then add one drop in excess. Cool quickly in running water, then add at one stroke 15 ml. of saturated $HgCl_2$ solution. Allow to stand for 3 min.; then transfer with washing to a 600-ml. or 1000-ml. beaker containing 300 ml. of cold distilled water and 25 ml. of the $MnSO_4$ solution. Titrate with standard 0.04 *N* $KMnO_4$ solution, added very slowly while stirring constantly, until a permanent pink end point is obtained. A blank should also be determined. Calculate the iron to FeO.

Silica

59. Fuse 0.5000 g. of the dried (105 to 110 C.) sample in approximately 10 g. of fused $K_2S_2O_7$ or $Na_2S_2O_7$ in a vitreous silica or porcelain crucible. The usual precautions of a pyrosulfate fusion should be observed; that is, the flame should be turned very low at the start so that the pyrosulfate just melts, then the flame increased so that the pyrosulfate remains in a liquid condition, and finally the temperature gradually raised until the crucible reaches a dull red heat. The object of the slow heating is to retain the H_2SO_4, which is the effective part of the flux. Excessive fuming is to be avoided because it may result in "crawling" of the melt up the sides of the crucible, producing the difficultly soluble anhydrous $Cr_2(SO_4)_3$. The most refractory materials should be thoroughly fused in 1 to 1½ hr. Remove the crucible from the burner, and allow to cool in the air. The pyrosulfate melt will shrink from the crucible, and the main mass may be removed easily in one cake. Dissolve the cake and the small portion retained on the crucible walls in 200 ml. of hot diluted HCl (1:1), boiling until the solution acquires a clear, bright green color. Evaporate to dryness, and bake at not over 120 C. until free from fumes of HCl, which will require about 1 hr. Moisten with hot diluted HCl (1:1), then add about 25 ml. more acid, warm until all salts are in solution, and filter off the SiO_2. Evaporate the filtrate to dryness, bake, and filter again. Wash the SiO_2 thoroughly with diluted HCl (5 per cent). Ignite in a tared platinum crucible to constant weight in an electric muffle furnace or over a moderate blast, and weigh. Add two drops of diluted H_2SO_4 (1:1) and about 10 ml. of HF to the crucible. Evaporate, ignite, and weigh as before. The difference represents the amount of SiO_2 present in the sample. Should there be any appreciable residue left in the crucible, fuse with a small amount of pyrosulfate and add to the filtrate from the SiO_2.

NOTE.—Decomposition by means of perchloric acid is permissible where it has been found that the particular chrome ore or burned refractory is amenable to such treatment. High-alumina ores and hard burned refractories are generally difficult to get completely into solution with perchloric acid.[9]

Alumina

60. (*a*) *Method A: R_2O_3 Precipitated with NH_4OH.*—Combine the filtrates from the silica determination (Section 59) and dilute to exactly 500 ml. in a graduated flask. Take a 300-ml. aliquot portion and add 15 g. of NH_4Cl and 3 drops of methyl red solution (0.1 per cent). Heat the solution almost to boiling, and add slowly NH_4OH (1:1) until the indicator has changed to a yellow color. Boil for 1 or 2 min. to remove excess ammonia, and allow the precipitate to settle. Filter through an 11-cm. rapid filter paper. Do not wash. Reserve the filtrate *A* for the determination of CaO and MgO (Section 61). Transfer the paper and precipitate to the beaker in which the precipitation was made, add 50 ml. of HCl (20 per cent), and digest covered on the hot plate, shaking frequently until the precipitate is dissolved and the paper well macerated. Dilute, add methyl red, reprecipitate with NH_4OH (1:1), boil, and filter, exactly as before. Add this second filtrate *B* to the first filtrate for the subsequent determination of CaO and MgO (Section 61). Swab the beaker and rod, and wash the paper and precipitate with hot NH_4Cl (2 per cent). Place the paper and precipitate in a tared platinum crucible and dry. Carefully burn off the paper, and then blast at 1200 C. to constant weight (30 min. is generally sufficient). Cool in a desiccator, and weigh with the crucible covered by the lid. The ignited precipitate consists of Fe_2O_3, Al_2O_3, Cr_2O_3, and TiO_2. To obtain the percentage of Al_2O_3, subtract from the weight of ignited precipitate the FeO (Section 58) calculated to Fe_2O_3, plus the TiO_2 (Section 57) and the Cr_2O_3 (Section 55).

(*b*) *Method B (Optional): R_2O_3 Precipitated with Urea.*—Dilute the entire combined filtrates from the silica determination (Section 59) to about 400 ml., preferably in a 600-ml. beaker. Add 15 g. of NH_4Cl, then add NH_4OH slowly until a slight permanent cloudiness appears. Clear the solution by adding diluted HCl (1:1) drop by drop and then 2 to 3 drops in excess. Now add 6 g. of urea (Note). Bring to a boil and continue boiling gently on the back of a hot plate for 2 hr. after the first opalescence appears. The decomposition of the urea causes a gradual increase in the pH of the solution, which is uniform throughout, and precipitates the R_2O_3 group as the dense basic sulfates. The final pH value of the solution is about the same as when using NH_4OH and the proper indicator. Remove from the hot plate, and allow the precipitate to settle, keeping the solution warm. Filter through two dense filter papers folded together, and transfer the bulk of the precipitate to the papers. The basic sulfate precipitate occupies only about one third of the volume of the equivalent hydroxide and is, accordingly, easily transferred and washed. For transferring the precipitate, use NH_4Cl solution (1 per cent) made just alkaline to methyl red indicator with NH_4OH. The small amount of precipitate adhering to the sides of the beaker may be removed by thorough scrubbing with a rubber policeman, or better, by dissolving with diluted HCl (5 per cent)

[9] For a description of a procedure using perchloric acid, see T. R. Cunningham and T. R. McNeil, "Analysis of Chrome Ores," *Industrial and Engr. Chemistry*, Anal. Ed., Vol. 1, p. 70 (1929).

directed against the side of the beaker from a wash bottle. Heat gently, add one drop of methyl red indicator, and then diluted NH_4OH (10 per cent) until just alkaline. Transfer the small amount of precipitated hydroxide to the filter paper holding the basic sulfate precipitate, using the NH_4Cl solution (1 per cent) referred to above. Scrub the beaker, and then wash the combined precipitates 10 times with the above solution. Reserve the filtrate for the determination of CaO and MgO (Section 61), and complete the procedure as described in Paragraph (*a*).

NOTE.—The precipitate obtained with urea is fine grained and much smaller in volume than the gelatinous hydroxides obtained with NH_4OH. This permits rapid filtering and more thorough washing.[10]

Lime

61. (*a*) Make the combined filtrates *A* and *B* from Section 60 (*a*), or the filtrate from Section 60 (*b*), slightly acid with HCl, evaporate to a volume of approximately 150 ml., and cool. Add 20 ml. of diammonium phosphate ($(NH_4)_2HPO_4$) solution (10 per cent), and slowly introduce NH_4OH drop by drop, while stirring vigorously, until the solution is ammoniacal and a crystalline precipitate appears. Add 15 ml. of NH_4OH, stir thoroughly, and allow to stand overnight at room temperature. Filter the solution on a 9-cm. filter paper, and wash the precipitate three or four times by decantation with cold NH_4OH (2.5 per cent). Pour 25 ml. of HCl (1:1) through the filter paper, collecting the filtrate in the original beaker containing the bulk of the precipitate. Wash the paper thoroughly with HCl (5 per cent). Dilute the solution to a volume of 150 ml. with cold water, add 3 ml. of $(NH_4)_2HPO_4$ (10 per cent), and repeat the precipitation. Allow to stand several hours. When cool, filter, wash 10 to 12 times with cold NH_4OH (2.5 per cent), and ignite in a tared platinum crucible, first at a dull red heat until the carbon has been removed, and finally to constant weight at about 1100 C. (20 to 30 min. is generally sufficient). The pyrophosphates thus obtained contain all the calcium, magnesium, and any manganese present.

(*b*) Dissolve the ignited pyrophosphates in 10 ml. of H_2SO_4 (1:1), and evaporate to fumes of SO_3. Cool, and add 5 ml. of water and enough absolute ethyl or methyl alcohol to constitute 90 to 95 per cent of the total volume of liquids present. Stir vigorously for several minutes. Allow the $CaSO_4$ precipitate to settle for 2 or 3 hr., or overnight if preferred, filter on a 9-cm. paper, and wash free of H_2PO_4 with ethyl alcohol (80 per cent). Dissolve the precipitate from the paper with HCl (10 per cent), and wash with about 25 ml. of the acid. Dilute to about 150 ml. with hot water, make alkaline with NH_4OH, heat to boiling, and precipitate the calcium by slowly adding 10 to 15 ml. of a saturated solution of ammonium oxalate. Digest on the hot plate just below boiling for 30 min. Filter on a 9-cm. paper, and wash with cold water until free from oxalate. Transfer the paper and precipitate to the original beaker, and dissolve with 150 ml. of H_2SO_4 (5 per cent). Heat almost to boiling, and titrate with 0.04 *N* $KMnO_4$.

Manganese as MnO

62. (*a*) Evaporate the alcoholic filtrate from the $CaSO_4$ (Section 61) until strong fumes of SO_3 are evolved and all organic matter is destroyed. After the

[10] For a description of an investigation of this procedure, see Hobart H. Willard and Ning Kang Tang, "Quantitative Determination of Aluminum by Precipitation with Urea," *Industrial and Engr. Chemistry*, Anal. Ed., Vol. 9, pp. 357 to 363 (1937)

solution has cooled, dissolve the residue in 25 ml. of HNO_3 (30 per cent). The oxidation of the manganese from this point may be effected by either of the following two methods:

Persulfate Method.—Add 75 ml. of water, 6 ml. of $AgNO_3$ solution (0.5 per cent), and 10 ml. of $(NH_4)_2S_2O_8$ solution. Boil for 1 min. and cool to room temperature.

Bismuthate Method.—If there is any evidence of brown hydrated MnO_2 after solution in the HNO_3, add H_2SO_3 drop by drop. Boil the solution, filter if necessary, cool to room temperature or lower, and add 2 to 3 g. of sodium bismuthate. Agitate the solution for 1 min., and filter through asbestos (Note). Wash with HNO_3 (3 per cent) until the residue is free from permanganic acid, as determined by the absence of the pink color in the washings.

NOTE.—A good grade of asbestos, acid washed and specially prepared for Gooch crucibles, should be used.

(*b*) Titrate at once with standard sodium arsenite (1 ml. is equivalent to 0.0002 g. of Mn), the permanganic acid formed from either the persulfate or the bismuthate method (Paragraph (*a*)). The end point is the disappearance of the pink color. The factor for converting Mn to MnO is 1.291.

Magnesia

63. Calculate the CaO obtained in Section 61 to $Ca_3(PO_4)_2$ by multiplying by the factor 1.845, and the MnO determined in Section 62 to $Mn_2P_2O_7$ by multiplying by the factor 2.001. Deduct the sum of these two phosphates from the weight of the combined phosphates of calcium, manganese, and magnesium in Section 61. The difference is the weight of $Mg_2P_2O_7$. The factor for converting $Mg_2P_2O_7$ to MgO is 0.362.

Standard Method of Test for

PYROMETRIC CONE EQUIVALENT (P.C.E.) OF REFRACTORY MATERIALS[1]

A.S.T.M. Designation: C 24 - 42

ADOPTED, 1920; REVISED, 1928, 1931, 1933, 1935, 1942.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 24; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test covers a procedure for determining the pyrometric cone equivalent (P.C.E.) of fire clay, fireclay brick, and silica cement by comparison of test cones with Standard Pyrometric Cones under the conditions herein prescribed.

Preparation of Sample

2. (*a*) *Clay or Brick.*—The entire sample of fire clay or fireclay brick, in case the amount is small, shall be crushed by means of rolls or a jaw crusher so as to produce a particle size not larger than $\frac{1}{4}$ in. In case the amount is large, a representative sample obtained by approved methods shall be so treated. The sample shall then be mixed thoroughly and the amount reduced to about 250 g. (0.5 lb.) by quartering. Any magnetic material introduced by crushing or grinding shall be removed by means of a magnet. This portion shall then be ground in an agate, porcelain, or hard steel mortar and the amount reduced again by quartering. The final size of the sample shall be 50 g. and the fineness such that it will pass a No. 70 (210-micron) A.S.T.M. sieve[3] (equivalent to mesh No. 65 of the Tyler standard series). In order to avoid excessive reduction of fines, they shall be removed frequently during the process of reduction by throwing the sample on the sieve and continuing the grinding of the coarser particles until all the sample passes through the sieve.

(*b*) *Silica Cements.*—In the case of silica cements, the sample obtained by approved methods shall be tested as received without grinding or other treatment.

NOTE.—For the purpose of this test, silica cement is understood to be the usual finely ground mixture of quartzite, silica brick, and fire clay of various proportions, and does not include materials sold under the name of high-temperature mortars.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-8 on Refractories.

[2] Prior to adoption as standard, this method was published as tentative from 1919 to 1920. Editorially revised in 1943.

[3] Detailed requirements for this sieve are given in the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

A "tentative revision" of this standard appears in the Tentative Revisions of Standards section at the back of this book.

Preparation of Test Cones

3. (*a*) The dried sample shall be thoroughly mixed and after the addition of sufficient dextrine, glue, gum tragacanth, or other alkali-free organic binder and water, shall be formed in a truncated trigonal pyramid with its base at a small angle to the trigonal axis, and of the following dimensions: Over-all height (between planes perpendicular to the trigonal axis) $1\frac{1}{8}$ in.; length of side of the truncated tip, $\frac{1}{32}$ in.; length of the two longer sides of the base, $\frac{9}{32}$ in.; base at an external angle of 82 deg. to one face and perpendicular to the symmetry plane through the opposite edge of the pyramid.

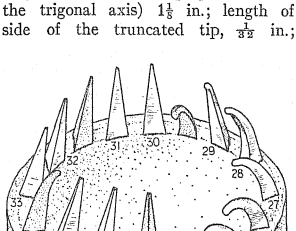


FIG. 1.—Method of Mounting Test Cones and Appearance After Testing.

(*b*) When dry the test cones may be subjected, when necessary, to a preliminary burn at a temperature not exceeding 2370 F. (1300 C.) for the purpose of sintering them to a firm condition to permit handling.

Mounting

4. The test cones shall be mounted on plaques of refractory material of such a composition as will not affect the fusibility of the cones.[4] They shall be mounted with the base embedded approximately 3 mm. (0.12 in.) in the plaque and the face of one side inclined at an angle of 82 deg. with the horizontal. The arrangement of the test cones with respect to the Standard Pyrometric Cones shall be substantially as shown in Fig. 1, that is, alternating the test cones with the Standard Pyrometric Cones in so far as is practical (Note). The plaque may be any convenient size and shape and may be biscuited before using, if desired.

NOTE.—The number of cones and their mounting so as to face inward as shown in Fig. 1 is typical for gas-fired furnaces of relatively large dimensions and gases moving at high velocity. The practical bore of the muffle tubes in most electric furnaces does not permit cone pats of this size. The static atmosphere prevailing permits the cones being mounted to face outward, if so desired.

Heating

5. (*a*) The heating shall be carried out in a suitable furnace at a rate to conform to the following requirements:

To reach cone No. 20	45 min.
Time interval to:	
Cone No. 23	20 min.
Cone No. 26	6 min.
Cone No. 27	4 min.
Cone No. 28	4 min.
Cone No. 29	10 min.
Cone No. 30	4 min.
Cone No. 31	12 min.
Cone No. 32	8 min.
Cone No. 32½	9 min.
Cone No. 33	9 min.
Cone No. 34	6 min.
Cone No. 35	10 min.
Cone No. 36	10 min.
Cone No. 37	4 min.
Cone No. 38	6 min.

NOTE.—At 1830 F. (1000 C.) the old cone pat may be removed and a new one put in without cooling the furnace to below red heat.

(*b*) That type of furnace in which a neutral or oxidizing atmosphere may be maintained is to be preferred. Exces-

[4] A mixture of equal parts of a highly refractory clay, such as a good grade of china clay and fused alumina, that will pass a No. 100 (149-micron) A.S.T.M. sieve (equivalent to mesh No. 100 of the Tyler standard series) has been found satisfactory.

sive reducing conditions should be avoided. Care should be taken that the flame does not strike directly against the cone or cone plaque. The furnace should be tested at intervals for the determination of the uniformity of the distribution of the heat.

Pyrometric Cone Equivalent

6. (*a*) The softening of the cone will be indicated by the top bending over and the tip touching the plaque. The bloating, squatting, or unequal fusion of small constituent particles should always be reported. The Pyrometric Cone Equivalent shall be reported in terms of Standard Pyrometric Cones and shall be that cone which most nearly corresponds in time of softening with the test cone. If the test cone softens later than one Standard Pyrometric Cone but earlier than the next Standard Pyrometric Cone and approximately midway between, the P. C. E. shall be reported thus: cone No. 31–32.

(*b*) If the test cone starts bending at an early cone but is not down until a later cone, the fact should be reported.

NOTE.—The temperatures corresponding to the end points of the Standard Pyrometric Cones are frequently of interest. These are shown in the Appendix.

APPENDIX

The temperatures corresponding to the end points of those Standard Pyrometric Cones that are used in connection with refractories are as follows:

Cone	End Point		Cone	End Point	
	Deg. Fahr.	Deg. Cent.		Deg. Fahr.	Deg. Cent.
No. 15	2615	1435	No. 30	3002	1650
No. 16	2669	1465	No. 31	3056	1680
No. 17	2687	1475	No. 32	3092	1700
No. 18	2714	1490	No. 32½	3131	1722 (approx.)
No. 19	2768	1520	No. 33	3173	1745
No. 20	2786	1530	No. 34	3200	1760
No. 23	2876	1580	No. 35	3245	1785
No. 26	2903	1595	No. 36	3290	1810
No. 27	2921	1605	No. 37	3308	1820
No. 28	2939	1615	No. 38	3335	1835
No. 29	2984	1640			

These temperatures (except cone No. 32½), which were determined by Fairchild and Peters[5] for a heating rate of 270 F. (150 C.) per hr. for cones Nos. 15 to 20 and of 180 F. (100 C.) per hr. for cones Nos. 23 to 38, other conditions being the same as specified, apply satisfactorily for all the conditions of this test method, but do not apply to conditions of the commercial burning and use of refractory materials.

[5] Fairchild and Peters, "Characteristics of Pyrometric Cones," *Journal*, Am. Ceramic Soc., Vol. 9, No. 11, p. 700, November, 1926.

Standard Method of

PANEL TEST FOR RESISTANCE TO THERMAL AND STRUCTURAL SPALLING OF REFRACTORY BRICK[1]

A.S.T.M. Designation: C 38 – 42

ADOPTED, 1936; REVISED, 1942.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 38; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. (*a*) This method of test outlines a general procedure for determining the resistance of refractory brick to the separate and combined effects of structural and thermal spalling. The test is believed to give a sufficient simulation of service conditions to suggest its use as a suitability test for certain classes of service that have been correlated with the test data.

(*b*) Detailed methods of test to be used in conjunction with this procedure in testing specific types of refractories are as follows:

Method of Panel Test for Resistance to Thermal and Structural Spalling of High Heat Duty Fireclay Brick (A.S.T.M. Designation: C 107),[3]

Method of Panel Test for Resistance to Thermal and Structural Spalling of Super Duty Fireclay Brick (A.S.T.M. Designation: C 122)[3], and

Method of Panel Test for Resistance to Thermal and Structural Spalling of Fireclay Plastic Refractories (A.S.T.M. Designation: C 180).[3]

Apparatus

2. The apparatus and equipment[4] shall consist of the following:

(*a*) *Panel Frame.*—A panel framework shall be used which is essentially the same as that shown in Fig. 1.

(*b*) *Preheating Furnace.*—A preheating furnace shall be used, constructed essentially like the design shown in Figs. 2 and 3.

(*c*) *Spalling Furnace.*—A spalling furnace shall be used, constructed in a manner as shown in Figs. 4, 5, and 6.

(*d*) *Temperature Measuring Instruments.*—The temperature of the test panel during the preheating shall be measured by means of an optical pyrometer. The spalling furnace temperature shall be determined by means of a platinum - platinum-rhodium thermocouple.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-8 on Refractories.

[2] Prior to adoption as standard, this method was published as tentative from 1934 to 1936. This method replaced the former water spalling method which was published as tentative from 1921 to 1934, being revised in 1926, 1927, 1931, and 1933, and which was accordingly discontinued in 1934.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

[4] The complete set of drawings necessary for the construction of the panel spalling equipment would require too much space to be included with the procedure, but these are available at a nominal charge from the Refractories Fellowship, Mellon Institute of Industrial Research, Pittsburgh, Pa.

A "tentative revision" of this standard appears in the Tentative Revisions of Standards section at the back of this book.

(*e*) *Air Supply.*—Means shall be provided for delivering air through each cooling unit at a specified rate.

(*f*) *Transfer Facilities.*—A suitable means, such as a track or trolley, shall be provided for transferring the test panels.

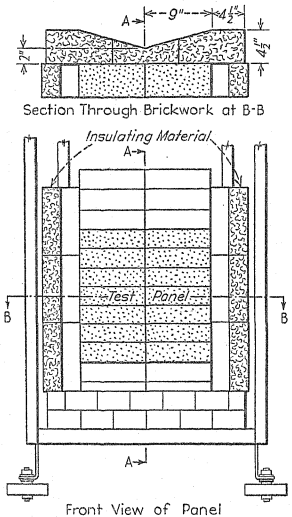


FIG. 1.—Panel Frame and Brickwork.
The brick around the test panel are dummy brick which are used to help maintain uniform conditions within the panel.

Test Specimens

3. Whenever possible, the specimens for the test panel shall consist of 9-in. straight brick. When shapes of other sizes are to be tested, the specimens shall be cut so as to be not more than 3 in. in thickness by not more than 9 in. in length by not more than $4\frac{1}{2}$ in. in width.

Panel Construction

4. The specimens shall be laid up so as to form a test panel not less than 18 in. square, so that a 9 by $2\frac{1}{2}$-in. surface of each specimen will be exposed to the heat treatment. Each specimen shall be labeled with ceramic paint on the face opposite that to be tested, and then weighed to the nearest 0.1 lb. There shall be placed about the test panel a suitable grade of refractory dummy brick, in the manner shown in Fig. 1. All brick in the test panel shall be laid up with refractory kaolin unless otherwise specified. The joints shall be not thicker than $\frac{1}{16}$ in. The test panel, including dummy brick, shall be insulated in accordance with the arrangement and design shown in Fig. 1.

Preheating Test Panels

5. The prepared test panels shall be placed in position on either side of the preheating furnace. When only one lot of brick is to be tested, a dummy panel should be used. The joint between the ends of the panel dummy brick and the preheating furnace shall be sealed with plastic kaolin. During the heating of the panels, the surface of the test specimen opposite that being heated shall be insulated in the specified manner so as to maintain a suitable temperature gradient in the specimen. The furnace shall be so operated that the temperature specified for preheating shall be reached in not less than 5 nor more than 8 hr. and maintained 24 hr. The permissible variation in temperature may be plus 35 F. and minus 45 F. (plus 20 C. and minus 25 C.), but the average of the temperature during the run shall be that specified. After completing the preheating, the test panel shall be left in position for not less than 8 hr. before being removed. As a result of this heat treatment, any tendency toward fusion, vitrification, swelling, or shrinkage will

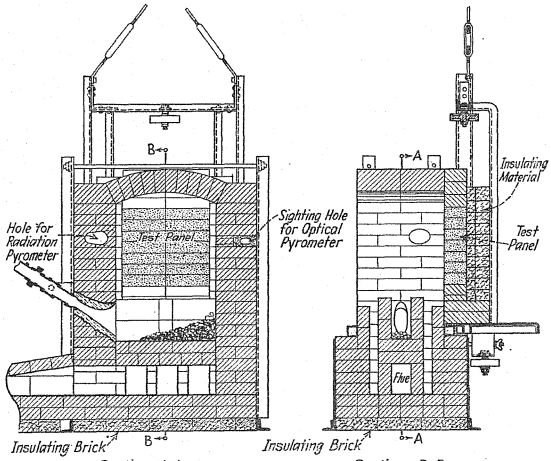


FIG. 2.—Design of Preheating Furnace.
In Section B-B there is shown in position one of the two panels used during the preheating.

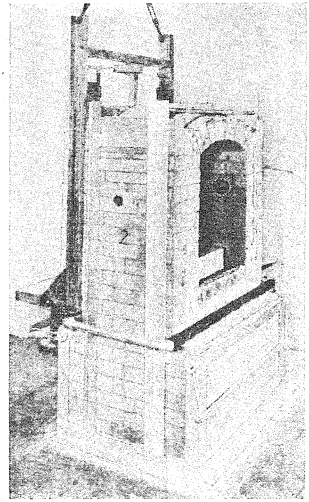


FIG. 3.—Preheating Furnace with Panel in Position on Far Side of Furnace.

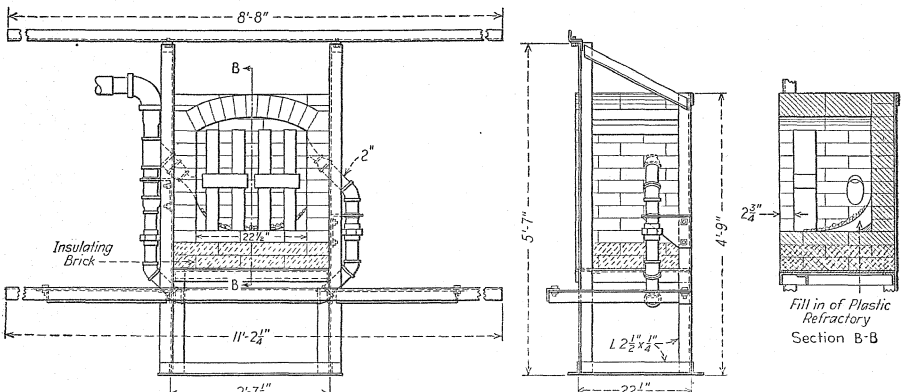


FIG. 4.—Furnace of the Spalling Equipment.
The complete layout is shown in Figs. 5 and 6.

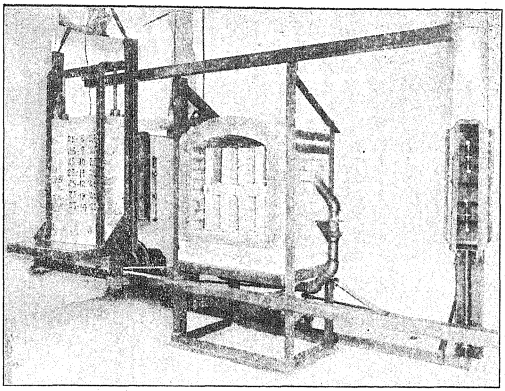


FIG. 5.—Spalling Equipment for Panel Spalling Test.

Front view showing one of the two panels in position. During the test a second panel is placed in front of the furnace opening. The two panels are linked together so that when the one which is now in the position for cooling is moved in front of the furnace, the other panel will be in position in front of the right-hand cooling unit. By this means, each panel is alternately heated and cooled.

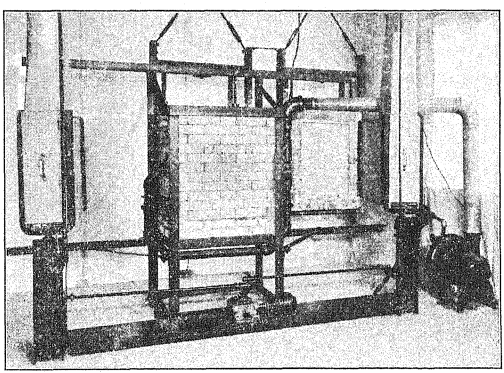


FIG. 6.—Cooling Equipment for Panel Spalling Test.

View from the back of the furnace showing the panels in position. There is shown the nature of the mechanism which causes the air manifolds to operate with a reciprocating motion and the motor blower unit used to operate the furnace.

be evident and any development of structural spalling will be apparent. A record shall be made of the condition of the brick specimens, indicating the extent of the fusion, vitrification, swelling,

Spalling Procedure

6. The test panels shall then be subjected to thermal spalling by heating them within a specified time to a specified temperature and rapidly cooling

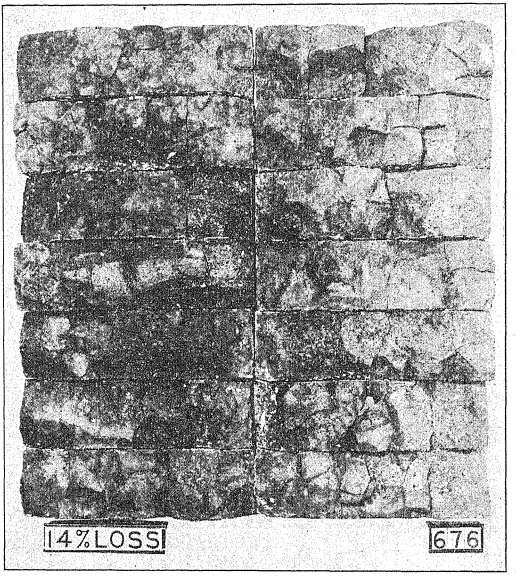


FIG. 7.—Photograph Showing Manner in Which Test Brick Are to Be Arranged for Making Photographic Report.
The brick are in the same corresponding position as laid up in the panel. The percentage loss and photograph number or designation are also shown.

or shrinkage. If shrinkage takes place a record shall be made of its nature, that is, whether or not this occurs as a shrinkage of each brick as a unit, or as the development of cracks on the surface of each brick. The surface of the test panels may be photographed.

by means of a prescribed blast. The panels shall be placed in position in front of the spalling furnace in the manner shown in Figs. 5 and 6. The temperature of the spalling furnace shall be raised within 3 hr. to the specified temperature. During the heating pe-

riod, the panels shall be alternately placed in position in front of the furnace so that each may be heated to about the same temperature. The position of the panels shall be changed at temperatures of 720, 360, and 180 F. (400, 200, and 100 C.) below the final furnace temperature. During this period, the test panels shall not be cooled by means of the cooling blast. After the final temperature of spalling is obtained, the panels shall be held in position for a specified time, and then shifted so as to expose the heated panel for cooling. After each panel has been subjected to the required number of cycles, the furnace shall be allowed to cool, but the cooling procedure for the test panels shall be conducted so that each panel receives two additional cycles without the water mist.

Dismantling Test Panel

7. When the test panel has cooled sufficiently so that the brick can be handled, the panel shall be carefully dismantled. Each of the test specimens shall be cleaned to free it of kaolin and in such a manner as to remove spalls, after which the specimens shall be reweighed to the nearest 0.1 lb., and the loss in weight recorded as percentage of the original weight. When a second heating treatment is given and the 9-in. brick specimens have been numbered from 1 to 7 from top to bottom in the test panel, their order shall be changed so as to assume the positions of 4–3–2–1–7–6–5. In the case of the six 3-in. brick specimens the order shall be changed so as to assume the positions of 3–2–1–6–5–4 for the second heating treatment. The specimens shall also be turned end for end.

Report

8. The report may include a photograph of the test panel after preheating, and shall include comments as to its condition. The average spalling loss for the whole panel shall be given, as well as a photograph of the spalled brick specimens arranged in the manner shown in Fig. 7.

Standard Method of

PANEL TEST FOR RESISTANCE TO THERMAL AND STRUCTURAL SPALLING OF HIGH HEAT DUTY FIRECLAY BRICK[1]

A.S.T.M. Designation: C 107 - 42

ADOPTED, 1936; REVISED, 1940, 1942.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 107; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test covers the procedure for determining the resistance of fireclay brick used for stationary boilers, malleable iron furnace bungs, and incinerator furnaces to the separate and combined effects of structural and thermal spalling. This method makes use of the general requirements and procedure prescribed in the Method of Panel Test for Resistance to Thermal and Structural Spalling of Refractory Brick (A.S.T.M. Designation: C 38) of the American Society for Testing Materials,[3] and supplements that method by giving the detailed requirements of the procedure to be followed in testing refractories for use in stationary boilers, malleable iron furnace bungs, and incinerator furnaces.

Apparatus and Test Specimens

2. The apparatus and test specimens shall be in general as described in the Method of Panel Test for Resistance to Thermal and Structural Spalling of Refractory Brick (A.S.T.M. Designation: C 38) of the American Society for Testing Materials.[3]

Procedure

3. The procedure shall be in general as outlined in Method C 38,[3] supplemented by the procedure for preheating and spalling as prescribed in the following Sections 4 and 5.

Preheating Test Panels

4. (*a*) The test brick shall be laid up with kaolin having a pyrometric cone equivalent not below cone No. 34.

(*b*) The test panels and dummy brick shall be insulated with group 26 insulating fire brick weighing 46 ± 2 lb. per cu. ft.

(*c*) The temperature of the panel faces shall be maintained so as to average 2910 F. (1600 C.) by means of a gas-fired furnace for a period of 24 hr.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-8 on Refractories.

[2] Prior to adoption as standard, this method was published as tentative from 1934 to 1936.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

A "tentative revision" of this standard appears in the Tentative Revisions of Standards section at the back of this book.

(*d*) The furnace gas pressure during the 24-hr. preheating period shall be within the limits of 0.3 to 0.6 in. of water for gas-fired furnaces.

NOTE.—In furnaces used for the purpose of obtaining control and experimental data where the method is followed in all details except that oil is used as a fuel, it is recommended that the furnace gas pressure be maintained between 0.1 and 0.2 in. of water.

Spalling Procedure

5. (*a*) The panel insulation shall be removed.

(*b*) The gas-fired furnace shall be operated so as to obtain a temperature of 2550 F. (1400 C.) before the panels are cooled by means of the blast.

(*c*) The period for heating shall be 10 min. and for cooling 10 min., making a 20-min. cycle.

(*d*) The cooling shall be accomplished by means of an air-water mist blast, which shall be delivered through a vertical manifold with an opening measuring $3\frac{1}{2}$ by 24 in. as shown in Method C 38.[3] Air shall be delivered through each cooling unit at the rate of 1400 cu. ft. per min. The water used in the four spray units shall be filtered and measured by means of a meter. The spray nozzles shall be regulated so as to deliver water at the rate of $2\frac{1}{2}$ gal. during the first 8 min. of the cycle. No water shall be used during the last 2 min. The cooling units shall be operated with a reciprocating motion so as to pass back and forth across the surface of the entire panel, including the dummy guard brick, 125 times in 10 min.

(*e*) The spalling treatment shall consist of 12 cycles, after which the furnace shall be shut off and the panels given two additional cooling cycles without the water mist.

(*f*) After carefully dismantling the panel, the kaolin shall be scraped off with a trowel, removing such pieces of the brick as separate easily. No attempt shall be made to separate pieces that might be removed by prying or tapping.

Standard Method of

PANEL TEST FOR RESISTANCE TO THERMAL AND STRUCTURAL SPALLING OF SUPER DUTY FIRECLAY BRICK[1]

A.S.T.M. Designation: C 122 - 42

ADOPTED, 1937; REVISED, 1940, 1942.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 122; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test covers the procedure for determining the resistance of super duty fireclay brick to the separate and combined effects of structural and thermal spalling. This method makes use of the general requirements and procedure prescribed in the Method of Panel Test for Resistance to Thermal and Structural Spalling of Refractory Brick (A.S.T.M. Designation: C 38) of the American Society for Testing Materials,[3] and supplements that method by giving the detailed requirements of the procedure to be followed in testing super duty fireclay brick.

Apparatus and Test Specimens

2. The apparatus and test specimens shall be in general as described in the Method of Panel Test for Resistance to Thermal and Structural Spalling of Refractory Brick (A.S.T.M. Designation: C 38) of the American Society for Testing Materials.[3]

Procedure

3. The procedure shall be in general as outlined in Method C 38,[3] supplemented by the procedure for preheating and spalling as prescribed in the following Sections 4 and 5.

Preheating Test Panels

4. (*a*) The test brick shall be laid up with kaolin having a pyrometric cone equivalent not below cone No. 34.

(*b*) The test panels and dummy brick shall be insulated with group 26 insulating fire brick weighing 46 ± 2 lb. per cu. ft.

(*c*) The temperature of the panel faces shall be maintained so as to average 3000 F. (1650 C.) by means of a gas-fired furnace for a period of 24 hr.

(*d*) The furnace gas pressure during the 24-hr. preheating period shall be within the limits of 0.3 to 0.6 in. of water for gas-fired furnaces.

NOTE.—In furnaces used for the purpose of obtaining control and experimental data where the method is followed in all details except that oil is used as a fuel, it is recommended that the furnace gas pressure be maintained between 0.1 and 0.2 in. of water.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-8 on Refractories.

[2] Prior to adoption as standard, this method was published as tentative from 1936 to 1937.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

A "tentative revision" of this standard appears in the Tentative Revisions of Standards section at the back of this book.

Spalling Procedure

5. (*a*) The panel insulation shall be removed.

(*b*) The gas-fired furnace shall be operated so as to obtain a temperature of 2550 F. (1400 C.) before the panels are cooled by means of the blast.

(*c*) The period for heating shall be 10 min. and for cooling 10 min., making a 20-min. cycle.

(*d*) The cooling shall be accomplished by means of an air-water mist blast, which shall be delivered through a vertical manifold with an opening measuring 3½ by 24 in. as shown in Method C 38.[3] Air shall be delivered through each cooling unit at the rate of 1400 cu. ft. per min. The water used in the four spray units shall be filtered and measured by means of a meter. The spray nozzles shall be regulated so as to deliver water at the rate of 2½ gal. during the first 8 min. of the cycle. No water shall be used during the last 2 min. The cooling units shall be operated with a reciprocating motion so as to pass back and forth across the surface of the entire panel, including the dummy guard brick, 125 times in 10 min.

(*e*) The spalling treatment shall consist of 12 cycles, after which the furnace shall be shut off and the panels given two additional cooling cycles without the water mist.

(*f*) After carefully dismantling the panel, the kaolin shall be scraped off with a trowel, removing such pieces of the brick as separate easily. No attempt shall be made to separate pieces that might be removed by prying or tapping.

Standard Method of Test for

PERMANENT LINEAR CHANGE AFTER REHEATING OF REFRACTORY BRICK[1]

A.S.T.M. Designation: C 113 - 36

ADOPTED, 1936.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 113; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test covers a procedure for determining the permanent linear change of certain types of refractory materials when heated under prescribed conditions.

Apparatus

2. The apparatus shall consist of a kiln of the down-draft type and of such design as not to permit the flame to impinge directly from the burner upon the test specimens. The kiln atmosphere during the test shall be kept as oxidizing as is practicable.

Test Specimens

3. (*a*) The test specimens shall consist of three standard 9-in. brick (9 by 4½ by 2½ in.) or three pieces of these dimensions cut out of larger shapes.

(*b*) Each test specimen shall be labeled with ceramic paint and carefully measured for length before and after reheating. The measurements shall be made by the use of a firm joint caliper and a steel rule. The calipering shall be done at the center of each end, and the dimension shall be measured to the nearest 0.02 in.

Location of Test Specimens in Kiln

4. The test specimens shall be placed in the kiln so that each will rest on a 9 by 2½-in. face and only one course high. Each specimen shall be placed upon the 9 by 2½-in. face of a supporting brick which shall be from the same lot as the test specimen or at least of equal refractoriness. There shall be placed between the test specimen and the supporting brick a layer of calcined diaspore, cyanite, or fused alumina ground to pass a No. 20 (840-micron) A.S.T.M. sieve (equivalent to mesh No. 20 of the Tyler standard series). Each specimen shall be placed so as to be not closer than 1½ in. from other test specimens or the furnace wall.

Temperature Measurement

5. (*a*) The temperature within the kiln shall be measured by means of a

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-8 on Refractories.

[2] Prior to adoption as standard, this method was published as tentative from 1934 to 1936. Editorially revised in 1942.

platinum - platinum-rhodium thermocouple and a pyrometer, preferably a recording pyrometer. The hot junction of the couple shall be placed so as to register the temperature of the test specimens. The kiln shall be of uniform temperature over the hearth and shall be checked from time to time by means of cones and shall not show a difference of more than one cone. Above 2640 F. (1450 C.) the thermocouple should be

Test Temperature Schedules

6. The kiln shall be operated so as to conform to the temperature schedule specified for the particular test to be conducted. Heating schedules for various classes of refractories are shown in Table I.

Cooling

7. After completion of the heating period the test specimens shall be allowed

TABLE I.—HEATING SCHEDULE FOR REHEAT TEST OF VARIOUS TYPES OF REFRACTORIES.[a]

Elapsed Time from Start of Heating	Temperature of Test Specimen			
	Schedule A	Schedule B	Schedule C	Schedule D
1 hr.....	between 1290 and 1470 F. (700 and 800 C.)	between 1650 and 1830 F. (900 and 1000 C.)	between 1650 and 1830 F. (900 and 1000 C.)	between 1515 and 1695 F. (825 and 925 C.)
2 hr.....	between 1965 and 2055 F. (1075 and 1125 C.)	between 2325 and 2415 F. (1275 and 1325 C.)	between 2325 and 2415 F. (1275 and 1325 C.)	between 2235 and 2325 F. (1225 and 1275 C.)
3 hr..	2190 ± 20 F. (1200 ± 10 C.) maintained for 5 hr.	2550 ± 20 F. (1400 ± 10 C.) maintained for 5 hr.	between 2640 and 2730 F. (1450 and 1500 C.)	2460 ± 20 F. (1350 ± 10 C.) maintained for 5 hr.
4 hr.....	.....	.....	between 2820 and 2865 F. (1550 and 1575 C.)	.....
5 hr.....	.....	.....	2910 ± 25 F. (1600 ± 15 C.) maintained for 5 hr.	.....

withdrawn and an optical pyrometer shall be used. The sighting shall be directed on one of the test specimens.

(*b*) During the constant temperature period of the test, the temperature as measured by the thermocouple shall not be allowed to vary by more than plus or minus 20 F. (10 C.) and in the case of the optical pyrometer, plus or minus 25 F. (15 C.) Temperature readings shall be made at intervals not greater than 15 min.

to cool in the closed kiln for a period of at least 10 hr.

Calculation

8. The test specimens shall be remeasured after rubbing the ends with an abrasive block to remove small blisters. The percentage linear change based upon the original length shall be calculated and reported as the average value of the three specimens.

[a] Editorially revised in 1942.

Standard Method of

TESTING FIRECLAY REFRACTORIES UNDER LOAD AT HIGH TEMPERATURES[1]

A.S.T.M. Designation: C 16 – 41

ADOPTED, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 16; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test covers the procedure for determining the resistance to deformation of fireclay refractories when subjected to a specified compressive load at a specified temperature for a specified time.

Apparatus

2. (*a*) The apparatus shall consist essentially of a furnace and a loading device. It may be constructed in accordance with Figs. 1, 2, or 3, or their equivalent.[3]

When gas or oil fuel is used, the furnace shall be of such form that the temperature is substantially uniform in all parts of the furnace. Two burners shall be used. These shall be capable of individual control and located to produce a tangential flame that will not impinge on the specimens or on the thermocouple protecting tubes.

(*b*) The temperature shall be measured either with calibrated platinum - platinum-rhodium thermocouples, each encased in a protection tube with the junction not more than 1 in. (25 mm.) from the center of the side or edge of each specimen farthest from the center of the furnace, or with some form of pyrometer that has been calibrated against a thermocouple in the furnace. A recording form of temperature indicator is recommended. If the optical pyrometer is used, observations shall be made by sighting into refractory tubes having closed ends and in the same relative positions to the test specimens as those specified for the thermocouples.

Test Specimen

3. (*a*) The test specimen shall consist of a minimum of two 9 by 4½ by 2½-in. straight refractory brick, or specimens of this size cut from larger shapes, utilizing as far as possible existing plane surfaces.

(*b*) If necessary, the ends of the

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-8 on Refractories.

[2] Prior to its present adoption as standard, this method was published as tentative from 1917 to 1920, being revised in 1920. It was adopted in 1920, published as standard from 1920 to 1939, being revised in 1936, but withdrawn and republished as tentative from 1939 to 1941. Editorially revised in 1942.

[3] Blueprints of detailed drawings of the furnaces shown in Figs. 1, 2, and 3 are available at a nominal cost from the American Society for Testing Materials, 260 S. Broad St., Philadelphia 2, Pa.

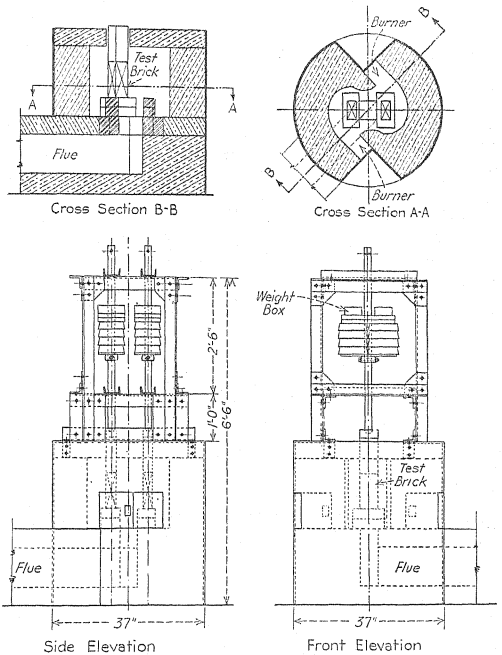


FIG. 1.—Direct-Load-Type Test Furnace.
The furnace is designed with a special contour for good heat distribution. It may be heated with either gas or oil.

(*c*) *Furnace Atmosphere.*—The furnace shall be operated with an oxidizing atmosphere above 1470 F. (800 C.).

(*d*) *Completion of Test.*—At the expiration of the time of heating, the supply of heat shall be stopped and the furnace allowed to cool by radiation to 1830 F. (1000 C.) or lower before the load is removed and the specimens examined. When the test specimens have cooled to room temperature, they shall be remeasured for length in accordance with Section 3(*c*), and the average deformation shall be recorded and reported to the nearest 0.5 per cent of the original length.

NOTE.—It is recommended that a photograph be made of the specimen before and after testing as yielding useful information.

Standard Methods of

TESTING INSULATING FIRE BRICK[1]

COMPRESSIVE STRENGTH, FLEXURAL STRENGTH, AND PERMANENT LINEAR CHANGE AFTER HEATING

A.S.T.M. Designation: C 93 - 42

ADOPTED, 1941; REVISED, 1942.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 93; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods of test cover procedures for determining the following properties of insulating fire brick:

Compressive strength,
Flexural strength, and
Permanent linear change as a result of heat treatment.

COMPRESSIVE STRENGTH

Apparatus

2. (*a*) Any universal testing machine[3] may be used.

(*b*) The spherical bearing block shall have a plane bearing surface, the area of which is equal to or greater than the cross-section of the test specimen.

Test Specimens

3. The test specimens shall consist of ten half-brick ($4\frac{1}{2}$ by $4\frac{1}{2}$ by $2\frac{1}{2}$ in.). They shall be placed on the $4\frac{1}{2}$ by $4\frac{1}{2}$-in. surfaces for test. The test surfaces of the specimens shall be approximately parallel planes.

Procedure

4. (*a*) The load shall be applied to the $4\frac{1}{2}$ by $4\frac{1}{2}$-in. surface of the test specimen.

(*b*) The bearing block shall be used on top of the test specimen in vertical testing machines. The bearing block shall be placed on the specimen so that the center of the sphere is in alignment with the vertical axis of the specimen (Fig. 1). The spherical bearing block shall be kept thoroughly lubricated to insure accurate adjustment, which should be made by hand under a small initial load.

(*c*) The testing machine shall be adjusted to apply the load at either of the following rates:

Hydraulic machine....	750 lb. per min. ± 10 per cent
Mechanical machine....	0.05 in. per min. ± 10 per cent

(*d*) During the test, when a mechanically actuated testing machine is used,

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[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee C-8 on Refractories.

[2] Prior to adoption as standard, these methods were published as tentative from 1933 to 1941, being revised in 1934 and 1939.

[3] The use of a hydraulic testing machine is recommended in preference to the mechanical type.

the beam of the testing machine shall be kept constantly in a floating position.

Calculation

5. The compressive strength in pounds per square inch shall be reported as calculated from the following formula:

$$S = \frac{W}{A}$$

where:

S = compressive strength in pounds per square inch,

W = load in pounds at 3 per cent deformation[4] or at visible failure, whichever is smaller, and

A = average of the gross areas of the top and bottom surfaces of the specimen in square inches.

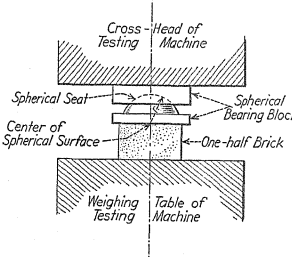


FIG. 1.—Spherical Bearing Block for Compressive Strength Test.

FLEXURAL STRENGTH

Apparatus

6. (*a*) Any universal testing machine[3] may be used.

(*b*) The bearing edges shall be $1\frac{1}{4}$ in. in diameter or rounded to a $\frac{5}{8}$-in. radius. They shall be straight and have full contact with the specimen and shall have a length at least equal to the width of the specimen to be tested. Their bases shall be so curved or rounded as to permit free movement of the working edges. The use of bearing cylinders of the type shown in Fig. 2 is recommended,

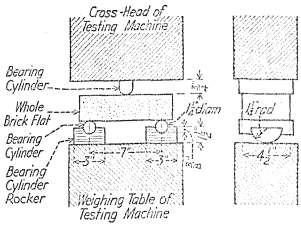


FIG. 2.—Recommended Design of Bearing Cylinders and Rockers for Flexural Strength Test.

although other types may be used, if they provide free movement and at the same time ensure full and even bearing at all times during the test. A satisfactory alternate design is shown in Fig. 3.

Test Specimens

7. At least ten test specimens shall be used and these shall each be a whole 9-in. brick (9 by $4\frac{1}{2}$ by $2\frac{1}{2}$ in.).

Procedure

8. (*a*) The bearing edges shall be placed so as to provide a 7-in. span. A test specimen shall be placed flatwise on the bearing edges and the load applied at mid-span.

(*b*) The testing machine shall be adjusted to apply the load at either of the following rates:

Hydraulic machine.....	200 lb. per min. ± 10 per cent
Mechanical machine....	0.05 in. per min. ± 10 per cent

[4] A dial-type micrometer is generally used for this measurement.

Calculation

9. The flexural strength in pounds per square inch shall be reported as calculated from the following formula:

$$R = \frac{3WL}{2bd^2}$$

where:

R = flexural strength in pounds per square inch,
W = load in pounds at which the specimen failed,
L = distance between the supports in inches,
b = width of the specimen in inches, and
d = depth of the specimen in inches.

(9 by 4½ by 2½ in.). If a specimen of some other size is used, the dimensions shall be reported.

Procedure

13. (*a*) Each test specimen shall be measured along its three major axes through the center. The measurements shall be made to the nearest 0.02 in.

(*b*) The test specimens shall be placed in the furnace preferably on the 9 by 2½-in. edge, resting on a slab of the same composition whose thickness shall be not less than 1 in. The test specimens shall be protected from the direct flame and from localized heating from direct contact with furnace parts.

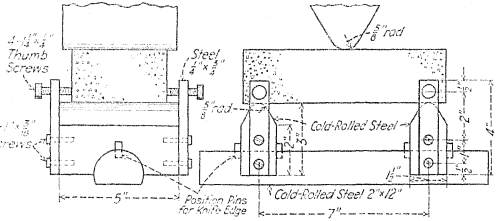


FIG. 3.—Recommended Design of Alternate Apparatus for Flexural Strength Test.

PERMANENT LINEAR CHANGE AFTER HEATING

Purpose

10. The object of this test is to determine the permanent linear change after heating of insulating fire brick when subjected to a definite temperature.

Apparatus

11. Any furnace in which uniform temperatures can be obtained may be used for the test.

Test Specimens

12. Three test specimens shall be used, these preferably being a whole 9-in. brick

(*c*) The maximum temperature to be used shall depend upon the material. This maximum temperature shall be obtained in not less than 3 hr. The maximum temperature shall be held for a period of 24 hr. The specimens shall be left in the furnace until the furnace temperature has fallen at least to 500 F. (260 C.).

Calculation

14. (*a*) *Permanent Linear Change.*—The permanent linear change shall be calculated from measurements taken along the three major axes of the specimens. The change in length, width, and thickness shall be reported separately. The permanent linear change shall be

calculated on the basis of each original dimension.

(*b*) *Permanent Volume Change.*—The permanent volume change shall be calculated from the measurements taken along the three major axes of the specimen in accordance with the following formula:

$$V = \frac{V_o - V_f}{V_o} \times 100$$

where:

V = percentage volume change,
V_o = original volume, and
V_f = final volume.

Standard Methods of Test for

COLD CRUSHING STRENGTH AND MODULUS OF RUPTURE OF REFRACTORY BRICK AND SHAPES[1]

A.S.T.M. Designation: C 133 - 39

ADOPTED, 1939.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 133; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods cover procedures for the following tests of refractory brick and shapes: Cold crushing strength and modulus of rupture.

COLD CRUSHING STRENGTH

Apparatus

2. (*a*) *Testing Machine.*—Any form of standard mechanical or hydraulic compression testing machine may be used.

(*b*) *Spherical Bearing Block.*—The spherical bearing block shall have a plane bearing surface, the area of which is equal to or greater than the cross-section of the test specimen.

Test Specimens

3. The test specimens shall be whole standard 9-in. brick (9 by 4½ by 2½ in.) or specimens of equivalent size ground or cut from refractory shapes. In the case of special shapes, only one specimen shall be cut from a single shape and as many of the original surfaces as possible shall be preserved. In preparing specimens from irregular or large refractory shapes, any method involving the use of abrasives, such as high-speed abrasion wheel or rubbing bed, that will produce a specimen with approximately plane and parallel sides without weakening the structure of the specimen may be used.

Preparation of Test Specimen

4. (*a*) The specimen to be used for the cold crushing strength test shall be prepared as follows: The two end surfaces (4½ by 2½ in.) intended to receive the load from the testing machine shall receive a thin coating of shellac and be allowed to dry thoroughly. One of the shellacked bearing surfaces shall then receive a thin coat of a neat mortar of calcined gypsum (plaster of Paris). This mortared face of the specimen shall be pressed on plate glass or other plane, nonporous surface which has been previously oiled or covered with waxed paper, and the bedding material allowed to harden. This procedure shall be

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee C-8 on Refractories.

[2] Prior to adoption as standard these methods were published as tentative from 1937 to 1939.

repeated with the other shellacked surface. Care shall be taken that the opposite faces so coated with bedding material shall be parallel.

(*b*) When the top and bottom surfaces of the specimen are approximately plane and parallel, a cellulose fiber wall board 0.25 in. in thickness and extending 2 in. or more over the edges of the specimen may be substituted for the above method of preparation.

(*c*) The report shall state which type of bedding material is used.

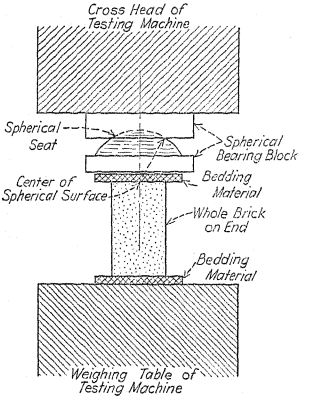


FIG. 1.—Recommended Design for Crushing Test Assembly, Including Bearing Block.

Procedure

5. (*a*) Five specimens shall be tested.

(*b*) The load shall be applied to the 4½ by 2½-in. ends of the test specimen.

(*c*) The bearing block shall be used on top of the test specimen in vertical testing machines. The center of the sphere of the bearing block shall be used in contact with the top bearing surface of the specimen and in the vertical axis of the specimen (Fig. 1). The spherical bearing block shall be kept thoroughly lubricated to insure accurate adjustment, which should be made by hand under a small initial load.

(*d*) The speed of head of the mechanical testing machine during the application of the load shall be not more than 0.5 in. per min. up to a pressure of 500 psi. and not exceeding 0.05 in. per min. for pressures over 500 psi. The rate of application of load, when using a hydraulic testing machine, shall be 20,000 lb. per min.[3]

(*e*) During the test, when a mechanically actuated testing machine is used, the beam of the testing machine shall be kept constantly in a floating position.

Calculation and Report

6. The cold crushing strength in pounds per square inch shall be reported as calculated from the following formula:

$$S = \frac{W}{A}$$

where:

S = cold crushing strength in pounds per square inch,
W = total maximum load, in pounds, indicated by the testing machine, and
A = average of the gross areas of the top and bottom of the specimen, in square inches, of the section of the specimen perpendicular to the line of application of the load

MODULUS OF RUPTURE

Apparatus

7. (*a*) *Testing Machine.*—Any form of standard testing machine may be used.

NOTE.—Portable apparatus properly calibrated may be used.

[3] This paragraph was editorially revised in 1940.

(*b*) *Bearing Edges.*—Bearing edges, $1\frac{1}{4}$ in. in diameter, shall be straight and have full contact with the specimen and shall have a length at least equal to the width of the brick to be tested. Their bases shall be so curved or rounded as to permit free movement of the working edges. The use of bearing cylinders of the type shown in Fig. 2 is recommended, although other types may be used which will provide free movement and at the same time insure full and even bearing at all times during the test. A satisfactory alternate design is shown in Fig. 3.

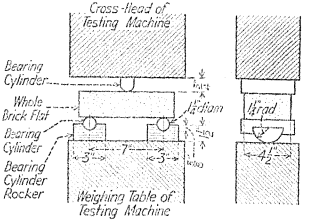


FIG. 2.—Recommended Design of Bearing Cylinders and Rockers for Use in Modulus of Rupture Test.

Test Specimens

8. The test specimens shall conform to the requirements prescribed in Section 3.

Procedure

9. (*a*) Five specimens shall be tested.

(*b*) A test specimen shall be placed flatwise on the bearing cylinders with a span of 7 in. and the load applied at mid-span.

(*c*) The speed of head of the mechanical testing machine during the application of the load shall be not more than 0.05 in. per min. The rate of application of load, when using a hydraulic testing machine, shall be 4000 lb. per min.[3]

Calculation and Report

10. The modulus of rupture in pounds per square inch shall be reported as calculated from the following formula:

$$R = \frac{3Wl}{2bd^2}$$

where:

R = modulus of rupture in pounds per square inch,

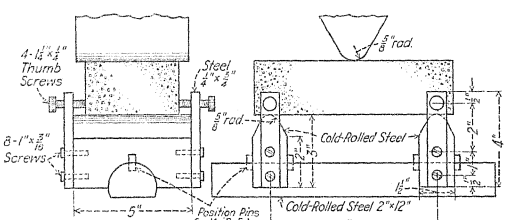


FIG. 3.—Recommended Design of Alternate Apparatus for Modulus of Rupture Test.

W = total load in pounds at which the specimen failed,

l = distance between the supports in inches,

b = width of the specimen in inches, and

d = depth of the specimen in inches.

Standard Methods of Test for
SIZE AND BULK DENSITY OF REFRACTORY BRICK[1]

A.S.T.M. Designation: C 134 - 41

ADOPTED, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 134; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods of test cover procedures for measuring the size and bulk density of rectangular refractory brick.

NOTE.—These methods include a procedure for measuring the size of rectangular refractory shapes.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Rule (12-in.).*—A steel rule 12 in. in length, graduated on one edge in $\frac{1}{8}\frac{1}{0}$ in. and and on the other in $\frac{1}{6}\frac{1}{4}$ in., for use in measuring brick and small shapes. The rule shall have a hook consisting of a right-angled piece on one end to fix the zero point of the scale against one face of the brick. The hook shall be $\frac{1}{4}$ in. in width and shall extend $\frac{1}{4}$ in. beyond the measuring side of the rule as shown in Fig. 1.

(*b*) *Rule (36-in.).*—A stiff steel hooked rule 36 in. in length, graduated in $\frac{1}{3}\frac{1}{2}$, $\frac{1}{6}\frac{1}{4}$, and $\frac{1}{1}\frac{1}{0}$ in. for use in stack measurements and for measuring the larger shapes by use of the hook. The 36-in. rule shall have the same design and the same graduations on the top of the rule as specified for the 12-in. rule, but on the other side and beginning at the opposite end from the hook it shall be graduated in $\frac{1}{3}\frac{1}{2}$ in.

NOTE.—The hooked rules shall be checked periodically to determine whether they have become distorted or worn in use.

(*c*) *Weighing Scale.*—A scale for weighing, having a capacity of 10 lb. or more and a sensitivity under load of at least 0.02 lb. ($\frac{1}{2}$ oz.)

Sampling

3. A sample of 20 brick or shapes shall be selected at random to represent each shipment, and 10 of these brick shall be taken as a primary set of test specimens. The remaining 10 brick shall be retained for retesting, if required.

Preparation of Specimens

4. Any pimples or fins shall be removed from the specimens by lightly rubbing them together.

Measurement of Size

5. (*a*) *Length and Width.*—The length and width of each of the 10 specimens shall be measured across the middle of

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee C-8 on Refractories.

[2] Prior to adoption as standard, these methods were published as tentative from 1938 to 1941, being revised in 1939.

each of the faces of largest area to the nearest $\frac{1}{8}$ in. or $\frac{1}{64}$ in. when the dimensions are 9 in. or under, and to the nearest $\frac{1}{2}$ or $\frac{1}{32}$ in. when the dimensions are over 9 in. The position of the hooked rule for these measurements is indicated by lines L and W in Fig. 2. The individual measurements of the two opposite faces of each specimen shall be made and recorded.

(*b*) *Thickness.*—The 10 specimens shall first be stacked on a plane surface without regard to the position of the brand marks on the specimens. The stack shall then be measured to the nearest $\frac{1}{32}$

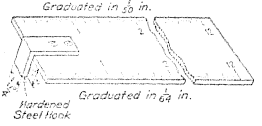


FIG. 1.—Design of 12-in. Hooked Rule.

in. from the plane surface to the top of the stack at the center of each side. The individual measurements of the four sides of the stack shall be recorded.

NOTE.—Brick or shapes over 3 in. in thickness shall be measured for thickness by the procedure described in Paragraph (*a*). The position of the hooked rule for the thickness measurements is indicated by the line T in Fig. 2.

Report

6. The average of the two individual measurements for length and for width of each specimen shall be reported. The thickness shall be reported as the average stack height divided by 10, except for those thickness measurements on brick over 3 in. in thickness obtained by the use of a hooked rule, which shall be reported as the average thickness of the individual specimens.

Retests

7. (*a*) If one specimen in the primary set of 10 specimens fails to conform to the length or width requirements specified, the length and width of the second set of 10 specimens shall be measured in accordance with Section 5 (*a*), and 19 of the 20 specimens must conform to the requirements specified.

(*b*) Failure of the stack thickness to conform to the requirements specified

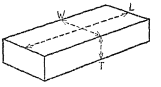


FIG. 2.—Measurements of Refractory Brick.

shall constitute cause for rejection. Thickness measurements obtained on individual specimens (that is, brick or shapes over 3 in. in thickness measured by use of the hooked rule) shall be subject to retest in accordance with Paragraph (*a*).

Bulk Density

8. Each of the specimens shall be weighed to the nearest 0.02 lb. ($\frac{1}{4}$ oz.), the weight recorded, and the average weight of the 10 specimens calculated. The average volume shall be calculated by using the average dimensions obtained in accordance with Section 6. The bulk density shall be calculated as follows:

$$\text{Bulk density, lb. per cu. ft.} = \frac{\text{wt. in lb.}}{\text{vol. in cu. in.}} \times 1728$$

Standard Method of Test for

WARPAGE OF REFRACTORY BRICK AND TILE[1]

A.S.T.M. Designation: C 154 – 41

ADOPTED, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 154; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test covers the procedure for determining the warpage of refractory brick and tile.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Steel Straightedge.*

(*b*) *Measuring Wedge.*—A steel wedge

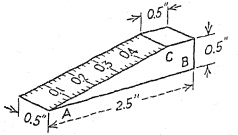


FIG. 1.—Measuring Wedge.

2.5 in. in length by 0.5 in. in width by 0.5 in. in thickness at one end, and tapered, starting at a line 0.5 in. from one end, to zero thickness at the other end. The wedge shall be graduated in 0.02 in. and numbered to show the thickness of the wedge between the base *AB* and the slope *AC*, as illustrated in Fig. 1.

Sampling[3]

3. The sample of brick or tile consisting of 20 pieces shall be taken at random to represent a shipment of a carload or fraction thereof.

Procedure

4. Place the straightedge across a diagonal on the concave side of the tile.

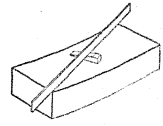


FIG. 2.—Method of Measuring Warpage.

Insert the wedge (Fig. 2) at the point of maximum warpage, and note the reading to the nearest 0.02 in. Repeat on the other diagonal.

Report[4]

5. The warpage shall be taken as the larger of the two measurements of each specimen. The report shall give the warpage values for the individual specimens.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-8 on Refractories.

[2] Prior to its present adoption as standard, this method was published as tentative from 1938 to 1939, constituting a portion of the Tentative Methods of Test for Size, Warpage, and Bulk Specific Gravity of Refractory Brick (C 134 – 38 T). This method was reissued in 1940 under the serial designation C 154, and published as tentative from 1940 to 1941.

[3] This section was added editorially in 1943 to correct an omission.

[4] This section was editorially revised in 1943.

Standard Methods of Test for

APPARENT POROSITY, WATER ABSORPTION, APPARENT SPECIFIC GRAVITY, AND BULK DENSITY OF BURNED REFRACTORY PRODUCTS[1]

A.S.T.M. Designation: C 20 – 41

ADOPTED, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 20; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods of test cover procedures for determining the following properties of burned refractory products:

Apparent porosity,
Water absorption,
Apparent specific gravity, and
Bulk density.

NOTE.—These methods are not applicable to materials attacked by water.

Preparation of Sample

2. (*a*) The sample shall consist of at least five standard 9-in. brick or similar units, from each of which a single test specimen, having a volume of approximately 25 or 26 cu. in. shall be cut or broken. When testing standard 9-in. straight brick, the specimen shall be a quarter-brick obtained by halving the brick along a plane parallel to the 9 by 2½-in. face and along a plane parallel to the 4½ by 2½-in. face. Four of the surfaces of the resultant quarter-brick specimen include part of the original molded faces. These surfaces are half of one end, half of one side and one quarter of each of the two 9 by 4½-in. faces. When testing large shapes, the sample shall consist of several specimens cut or broken from each shape from both the center and outer portions.

(*b*) Each specimen shall be freed of all loosely adhering particles.

(*c*) Visibly defective specimens shall not be used.

Dry Weight, *D*

3. (*a*) The test specimens shall be dried to constant weight by heating to 220 to 230 F. (105 to 110 C.) and the dry weight, *D*, in grams determined to the nearest 0.1 g.

(*b*) The drying procedure may be omitted only when the test specimens

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee C-8 on Refractories.

[2] Prior to their present adoption as standard, these methods were published as tentative from 1918 to 1920. They were adopted in 1920, published as standard from 1920 to 1939, being revised in 1931 and 1933, but withdrawn and republished as tentative from 1939 to 1941. Editorially revised in 1942 and 1943.

are known to be dry, as may be the case with samples taken directly from kilns.

(*c*) The drying of the specimens to constant weight and the determination of their dry weights may be done either before or after the boiling operation (Section 4). Usually the dry weight is determined before boiling; if, however, the specimens are friable or evidence indicates that particles have broken loose during the boiling operation, the specimens shall be dried and weighed after the suspended weight, *S*, and the saturated weight, *W*, have been determined as described in Sections 5 and 6. This second dry weight shall be used in all appropriate calculations.

Boiling

4. (*a*) The test specimens shall be placed in water and boiled for 2 hr. During the boiling period, they shall be kept entirely covered with water, and shall not be in contact with the heated bottom of the container.

(*b*) After the boiling period, the test specimens shall be cooled to room temperature while still completely covered with water. The cooling may be accelerated by running cold water into the container.

Suspended Weight, *S*

5. (*a*) The weight, *S*, of each test specimen after boiling and while suspended in water shall be determined in grams to the nearest 0.1 g.

(*b*) This weighing is usually accomplished by suspending the specimen in a loop or halter of 22-gage copper wire hung from one arm of the balance The balance shall be previously counterbalanced with the wire in place and immersed in water to the same depth as is used when the refractory specimens are in place.

Saturated Weight, *W*

6. After determining the suspended weight, each specimen shall be blotted lightly with a moistened smooth linen or cotton cloth to remove all drops of water from the surface and the saturated weight, *W*, determined in grams by weighing in air to the nearest 0.1 g. The blotting operation shall be performed by rolling the specimen lightly on the wet cloth, which has previously been saturated with water and then pressed only enough to remove such water as will drip from the cloth. Excessive blotting will introduce error by withdrawing water from the pores of the specimen.

Exterior Volume, *V*

7. The volume, *V*, in cubic centimeters of the test specimens may be obtained by subtracting the suspended weight from the saturated weight, both in grams, as follows:

$$V = W - S$$

NOTE.—This assumes that 1 cu. cm. of water weighs 1 g. This is true within about three parts in 1000 for water at room temperature.

Volumes of Open Pores and Impervious Portions

8. The volume of open pores and the volume of the impervious portions of the specimen may be calculated as follows:

Vol. of open pores in cu. cm. $= W - D$
Vol. of impervious portion in cu. cm. $= D - S$

Apparent Porosity, *P*

9. The apparent porosity expresses as a percentage the relationship of the volume of the open pores of the specimen to its exterior volume and shall be calculated as follows:

$$P = \frac{W - D}{V} \times 100$$

Water Absorption, *A*

10. The water absorption, *A*, expresses as a percentage the relationship of the weight of water absorbed to the weight of the dry specimen and shall be calculated as follows:

$$A = \frac{W - D}{D} \times 100$$

Apparent Specific Gravity, *T*

11. The apparent specific gravity, *T*, of that portion of the test specimen which is impervious to boiling water shall be calculated as follows:

$$T = \frac{D}{D - S}$$

Bulk Density, *B*

12. (*a*) The bulk density, *B*, in grams per cubic centimeter of a specimen is the quotient of its dry weight divided by the exterior volume, including pores and shall be calculated as follows:

$$B = \frac{D}{V}$$

(*b*) This method of determining bulk density is useful for checking bulk density values obtained by the direct measurement method[3] (Note). While it is more accurate than the direct measurement method, and generally gives higher values (by about 0.02 to 0.04), the direct measurement method is better suited for plant and field testing, since it is a less involved technique. The present method is preferable for specimens which are branded deeply or irregular in contour.

NOTE.[4]—The values for bulk density determined according to Section 12 will be expressed in metric units, for example, grams per cubic centimeter. The bulk density, however, determined by the direct measurement method will be expressed in English units. The conversion from metric to English units may be calculated as follows:

Bulk density in g. per cu. cm. × 62.43 = Bulk density in lb. per cu. ft.

Report

13. (*a*) For each property, the average of the values obtained with at least five specimens, and preferably also the individual values, shall be reported.[5]

(*b*) Apparent porosity and water absorption results shall be reported to one decimal place, and apparent specific gravity and bulk density results to two decimal places.

NOTE.—When values are reported for water absorption but not for porosity, it is suggested that the report shall also give the results for bulk density. This makes it possible to calculate the corresponding apparent porosity values as follows:

$$P = A \times B$$

[3] Methods of Test for Size and Bulk Density of Refractory Brick (A.S.T.M. Designation: C 134), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

[4] This note was added editorially in 1944.

[5] When there are pronounced differences among the individual values, another sample of five specimens shall be tested. These shall consist of the quarter-brick diagonally opposite from the original specimens. The average of all ten determinations shall be reported.

Standard Method of Test for

TRUE SPECIFIC GRAVITY OF BURNED REFRACTORY MATERIALS[1]

A.S.T.M. Designation: C 135 - 40

ADOPTED, 1940.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 135; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test covers a procedure for the determination of true specific gravity of refractory materials under prescribed conditions.

NOTE.—This method is not applicable to materials attacked by water.

Apparatus

2. The apparatus shall consist of the following: Analytical balance and weights, 50-ml. pycnometer bottle with capillary tube stopper, thermometer, drying oven, weighing bottle, desiccator, a suitable apparatus to produce a vacuum of 0.5 to 1.0 in. mercury pressure, and distilled water at room temperature.

Test Specimens

3. (*a*) The test specimen shall consist of two walnut-size pieces cut from different positions in the sample in such a way as to exclude all skin surfaces. If an average specific gravity is desired, specimens from at least five samples shall be tested.

(*b*) The specimen shall be crushed, if necessary, between hardened steel surfaces and then ground in an agate mortar, either by hand or by a mechanical sample grinder[3] so constructed as to prevent the introduction of any impurity. The specimen shall then be reduced to 50-g. by quartering in the standard manner. Any magnetic material introduced by crushing or grinding shall be removed by means of a magnet. The entire 50-g. sample shall be ground in an agate mortar so that it will pass a No. 100 (149-micron) A.S.T.M. sieve[4] (equivalent to mesh No. 100 of the Tyler standard series). Care shall be taken at all stages of the grinding and quartering not to exclude any portions which are difficult to grind, that is, all selective specimen preparation shall be avoided.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-8 on Refractories.

[2] Prior to adoption as standard, this method was published as tentative from 1938 to 1940. Editorially revised in 1942.

[3] The McKenna, Carling, Fisher, or similar type sample grinders are satisfactory for this purpose.

[4] Detailed requirements for this sieve are given in the Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Procedure

4. (*a*) The 50-g. sample shall be dried to constant weight at 220 to 230 F. (105 to 110 C.) and placed in a glass-stoppered weighing bottle. Duplicate tests shall be made on material from the 50-g. sample. All weights shall be recorded to the nearest 0.0001 g.

(*b*) The pycnometer and stopper shall be dried at 220 to 230 F. (105 to 110 C.) cooled in a desiccator, weighed on an analytical balance, and the weight recorded as weight p. The pycnometer shall then be filled with distilled water at room temperature, t, and again weighed with the stopper in place, recording the weight as W_1. The pycnometer shall then be emptied and again dried.

(*c*) Approximately 8 to 12 g. of the sample shall be placed in the dry pycnometer; the pycnometer, stopper, and sample shall be weighed, and the weight recorded as weight W. The bottle shall next be filled to approximately one-half its capacity with distilled water at room temperature t. The sample and water shall be very thoroughly mixed by stirring with a glass rod and evacuated (Note) at 0.5 to 1.0 in. mercury pressure to remove entrapped air.

NOTE.—A suitable method of evacuation is described in Section 4 (*e*) of the Methods of Test for Specific Gravity of Pigments (A.S.T.M. Designation: D 153) of the American Society for Testing Materials.[5]

(*d*) After evacuation, the bottle shall be filled with distilled water at room temperature t, and the excess water on the tip of the capillary removed by filter paper. The pycnometer and contents shall then be weighed and the weight recorded as weight W_2.

[5] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Calculation

5. (*a*) The true specific gravity shall be calculated in accordance with the following formula:

$$\text{Sp. gr., } t/t = \frac{W - p}{(W - p) - (W_2 - W_1)}$$

where:

t = temperature of the material and the water,
p = weight of the stoppered pycnometer,
W = weight of the stoppered pycnometer and sample,
W_1 = weight of the stoppered pycnometer filled with water, and
W_2 = weight of the stoppered pycnometer, sample, and water.

(*b*) The true density of the sample may be determined without additional measurement in accordance with the following formula:

$$\text{True density} = \text{sp. gr.} \times (d_w - d_a)$$

where:

d_w = density of water at the temperature at which the test was made, and
d_a = density of air at the temperature at which the test was made.[6]

Reproducibility of Results

6. Both determinations shall be reported to the nearest 0.001, and they shall check to within 0.005, or additional determinations shall be made.

[6] Values of $(d_w - d_a)$ for temperature from 15 to 30 C. are given in Table 35, Standard Density and Volumetric Tables, National Bureau of Standards, *Circular No. 19*, p. 52.

Standard Definitions of

TERMS RELATING TO REFRACTORIES[1]

A.S.T.M. Designation: C 71 - 42

ADOPTED, 1928; REVISED, 1931, 1935, 1936, 1942.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 71; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

MATERIALS

Bond Fire Clay.—See Plastic or Bond Fire Clay.

Diaspore Clay.—A rock consisting essentially of diaspore bonded by fire clay.

Fire Clay.—A sedimentary clay[3] of low flux content.

NOTE.—It is usually associated with coal measures.

Flint Fire Clay.—A hard or flint-like fire clay occurring as an unstratified massive rock, practically devoid of natural plasticity and showing a conchoidal fracture.

Grog Fireclay Mortar.—Raw fire clay mixed with calcined fire clay, or with broken fireclay brick, or both, all ground to suitable fineness.

Ground Fire Clay.—Fire clay or a mixture of fire clays that have been subjected to no treatment other than grinding or weathering, or both.

High-Temperature Bonding Mortar.—A mixture of refractory materials (such as fire clay, quartzite, chrome ore, magnesite, diaspore, or sillimanite minerals) either raw or calcined, to which other materials, not classified as refractories, have been added for the purpose of increasing the plasticity, giving air-setting properties, or lowering the temperature at which the bond develops.

Nodular Fire Clay.—A rock containing aluminous or ferruginous nodules, or both, bonded by fire clay.

NOTE.—In some districts such clays are called "burley" or "burley flint" clay.

Plastic or Bond Fire Clay.—A fire clay of sufficient natural plasticity to bond nonplastic materials.

Silica Fire Clay.—A refractory mortar consisting of a finely ground mixture of quartzite, silica brick, and fire clay of various proportions.

NOTE.—Sometimes called silica cement by the trade.

PROPERTIES

Abrasion of Refractories.—Wearing away of refractory surfaces by the scouring action of moving solids.

Burning (Firing) of Refractories.—The final heat treatment to which refractory brick and shapes are subjected in the

[1] Under the standardization procedure of the Society, these definitions are under the jurisdiction of the A.S.T.M. Committee C-8 on Refractories.

[2] Prior to adoption as standard, these definitions were published as tentative from 1927 to 1928, being revised in 1928.

[3] The definition for the term "fire clay" is based upon the following definition for the term "clay:"

Clay.—An earthy or stony mineral aggregate consisting essentially of hydrous silicates of alumina, plastic when sufficiently pulverized and wetted, rigid when dry, and vitreous when fired at a sufficiently high temperature.

A "tentative revision" of this standard appears in the Tentative Revisions of Standards section at the back of this book.

process of manufacture for the purpose of developing bond and other necessary physical and chemical properties.

Corrosion of Refractories.—Destruction of refractory surfaces by the chemical action of external agencies.

Erosion of Refractories.—Wearing away of refractory surfaces by the washing action of moving liquids.

Pyrometric Cone Equivalent (*P.C.E.*).—The number of that Standard Pyrometric Cone whose tip would touch the supporting plaque simultaneously with a cone of the refractory material being investigated when tested in accordance with the Standard Method of Test for Pyrometric Cone Equivalent (P.C.E.) of Refractory Materials (A.S.T.M. Designation: C 24) of the American Society for Testing Materials.

NOTE.—The terms "fusion point," "softening point," "deformation point," and "melting point" have heretofore been loosely used for "pyrometric cone equivalent."

Slagging of Refractories.—Destructive chemical reaction between refractories and external agencies at high temperatures, resulting in the formation of a liquid.

Spalling of Refractories.—The breaking or crushing of a refractory unit due to thermal, mechanical, or structural causes, presenting newly exposed surfaces of the residual mass.

NOTE.—The causes of spalling may be classified under three main headings, as follows:

Thermal:

(*a*) *Factors Related to Service.*—Rapidity and range of thermal fluctuation; contamination by slags and fluxes; tightness of joints; previous vitrification.

(*b*) *Factors Related to Refractories.*—Degree and uniformity of reversible thermal expansion; heat transfer; elasticity; plastic flow.

Mechanical:

(*a*) *Factors Related to Service.*—Rapid heating of wet brick; abuse in removing clinker and slag; unequal and excessive stresses; pinching; no provision for expansion; thin joints.

(*b*) *Factors Related to Refractories.*—Mechanical strength; toughness; accuracy of shape.

Structural:

(*a*) *Factors Related to Service.*—Slags and fluxes; character of the material in joints; insulation of refractories.

(*b*) *Factors Related to Refractories.*—Vitrification; shrinkage; nature of bond; structure; degree of burning.

Standard Methods of

CHEMICAL ANALYSIS OF GLASS SAND[1]

A.S.T.M. Designation: C 146 - 43

ADOPTED, 1943.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 146; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods for the chemical analysis of glass sand cover procedures for use in referee analysis. While primarily adapted for the analysis of glass sand containing not less than 98 per cent silica, these methods are not intended to preclude the use of any other method that yields results within permissible variations. In any case, the analyst should check the procedure and technique that is employed by means of a National Bureau of Standards' standard sample having a composition comparable with that of the material under test. A list of these standard samples is given in the Bureau's Supplement to *Circular 398*.

Special Solutions Required

2. (*a*) *Purity of Reagents.*—All reagents must be free from such amounts of impurities as would endanger the test in question. For referee analysis, reagents conforming to the specifications of the Committee on Analytical Reagents of the American Chemical Society are recommended.

(*b*) *Correction for Impurities in Reagents.*—Blank determinations on all reagents employed must be carried through each step of the procedure and the final result corrected as may be indicated.

(*c*) *Concentration of Acids.*—Throughout these methods the use of concentrated acids is intended, unless otherwise specified. For example, hydrochloric acid, or HCl, refers to the concentrated acid (sp. gr. 1.19) as furnished by the manufacturer. Diluted acids will be designated as in the following example: "diluted HCl (2:98)" will mean two volumes of the concentrated acid of sp. gr. 1.19, diluted with 98 volumes of distilled water.

(*d*) *Water.*—The term "water" as used in these methods refers only to distilled water.

Preparation of Sample

3. (*a*) The sample received for chemical analysis shall be passed through an 840-micron (No. 20) sieve.[3] Any por-

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee C-14 on Glass and Glass Products.

[2] Prior to adoption as standard, these methods were published as tentative from 1939 to 1943.

[3] Detailed requirements for this sieve are given in the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E11), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

tion of the sample that is retained on the sieve shall be crushed, without contamination, until it will pass through the sieve. The screened sample shall be thoroughly mixed and, if necessary, reduced in size by quartering. Not less than 1 lb. of the prepared sample shall be retained for use in the chemical analysis.

(*b*) If the well-mixed screened sample appears to be nonuniform, the sample for analysis shall be taken by spreading the well-mixed sample on a clean surface, flattening to a depth of approximately 0.5 in., and removing small portions at ten or more representative positions.

Loss on Ignition

4. Transfer 1.0000 g. of the sample, dried at 105 to 110 C., to a clean 25-ml. platinum crucible weighed with cover. Cautiously heat the crucible with contents, at first partially covered, and then at approximately 1000 C. covered. Cool in a desiccator, and weigh. Repeat the heating until constant weight is obtained. The loss in weight multiplied by 100 represents the percentage loss on ignition.

Silicon Dioxide

5. Transfer 0.5000 to 1.0000 g. of the sample, dried at 105 to 110 C., to a clean 25-ml. platinum crucible, add about four times as much anhydrous Na_2CO_3 as the weight of the sample taken, and mix thoroughly by stirring with a platinum rod or wire. Cover the crucible and fuse. When cool, transfer the crucible and melt to a large platinum dish, add water, cover, and digest. As soon as the melt has become detached, remove and thoroughly wash the crucible and cover. Acidify the water extract with diluted HCl (1:2), avoiding loss by effervescence. Evaporate to dryness on a steam or water bath. Cool, add 10 ml. of HCl, digest for 5 to 10 min., and dilute with 200 ml. of hot water. Digest until soluble salts are dissolved. Immediately filter through a S. & S. No. 589 black ribbon, or equivalent, paper; wash the SiO_2 and paper with small portions of hot diluted HCl (1:19) and finally with hot water. Transfer the paper and precipitate to a platinum crucible, weighed with cover, and reserve. Evaporate the filtrate to dryness, cover, and heat the dish and contents at 115 to 120 C. for 1 hr. Cool, dissolve the residue in HCl and water, filter, and wash as before to recover the SiO_2 that escaped the first dehydration. Reserve the filtrate if a further recovery of SiO_2 is contemplated. Transfer the paper and precipitate to the platinum crucible, moisten with a few drops of diluted H_2SO_4 (1:4), and ignite cautiously until the paper is charred. Partially cover the crucible, slowly increase the heat until the carbon has been burned, and then cover the crucible and ignite at approximately 1200 C. for 30 min. In all of the heating, great care must be taken to avoid loss of the powdery SiO_2 through drafts. Cool over a good desiccant and weigh. Repeat the ignition until constant weight is obtained. Moisten the contents of the crucible with 5 ml. of water, add 2 drops of diluted H_2SO_4 (1:1) and add (cautiously) 15 ml. of HF, evaporate to dryness, fume off the excess of H_2SO_4, and ignite at about 1000 C. Cool and weigh. If a 0.5-g. sample was taken, the percentage of SiO_2 is obtained by multiplying by 200 the difference between the original weight of the covered crucible and its contents and the final weight.[4]

[4] Any SiO_2 escaping the double dehydration may be recovered by adding approximately 0.05 g. of ferric iron as chloride or sulfate to the reserved filtrate, precipitating with NH_4OH, filtering, washing to remove sodium salts, drying, igniting at about 1100 C., cooling, and weighing in the customary manner. The residue thus obtained is treated with 5 ml. of HF and 10 drops of diluted H_2SO_4 (1:1), evaporated to dryness, ignited at about 1100 C., and weighed. The SiO_2 recovered, as indicated by the loss in weight, is added to that previously found. The recovery may amount to as much as 1 mg., which represents 0.1 and 0.2 per cent of SiO_2, respectively, for 1 and 0.5-g. samples.

Ferric Oxide

NOTE.—The following procedure is satisfactory for determining iron up to a total of 10 mg. of Fe_2O_3. For larger quantities the size of the sample should be so reduced that the 10 mg. is not exceeded. This may be accomplished by using a smaller sample of the sand, or preferably by taking an appropriate aliquot portion of the solution obtained after decomposition of the 10-g. sample.

6. (*a*) *Special Solutions Required: Potassium Dichromate (0.005 N).*—Recrystallize, three times, the purest grade of $K_2Cr_2O_7$ obtainable. Dry the crystals at 150 C., grind to a fine powder, and dry at 200 C. until constant weight is attained. Dissolve 2.4516 g. of the recrystallized salt in water and dilute to exactly 10 liters. One milliliter of exactly 0.005 *N* $K_2Cr_2O_7$ represents 0.000399 g. of Fe_2O_3.

Stannous Chloride Solution.—Dissolve 150 g. of $SnCl_2 \cdot 2H_2O$ in 100 ml. of HCl and dilute to 1000 ml. with water.

(*b*) *Procedure.*—Transfer 10 g. of the dried sand to a 150 to 200-ml. platinum dish. Moisten the sample with water, and add 5 ml. of diluted H_2SO_4 (1:1).[5] Add cautiously 100 ml. of HF, mix thoroughly by stirring with a platinum rod or spatula, and evaporate to 25 ml. on a steam or sand bath. Add 50 ml. of HF and evaporate to fumes of H_2SO_4. To the gently fuming solution add cautiously, drop by drop, a few drops of HNO_3 to destroy organic matter. Cool, rinse the walls of the dish with water, and again heat to strong fumes of H_2SO_4 in order to expel all HNO_3. Cool the dish and contents, add 100 ml. of water, heat to boiling, filter, and wash the residue and paper with hot water. Reserve the filtrate. Ignite the paper and insoluble matter, fuse with a small amount of Na_2CO_3, and dissolve the melt in diluted H_2SO_4 (1:5). Filter, wash the residue and paper, and combine the filtrate with that previously reserved. Again ignite the paper and insoluble matter. Fuse with a minimum quantity of potassium pyrosulfate ($K_2S_2O_7$), dissolve the melt in diluted H_2SO_4 (1:5) and combine with the reserved filtrates. Dilute or concentrate the combined filtrates so that the volume is approximately 200 ml., thoroughly saturate with H_2S, and digest, preferably overnight. Filter into a 500-ml. Erlenmeyer flask and wash the precipitate and paper with diluted H_2SO_4 (1:99) saturated with H_2S. Boil the filtrate to expel H_2S and reduce the volume to 100 ml. Oxidize any reducing substances that may be present by adding 0.1 *N* $KMnO_4$ until a permanent pink coloration is produced. Add 100 ml. of HCl, heat to boiling, and reduce the iron with the minimum amount of a solution of $SnCl_2$. Titrate electrometrically with standard 0.005 *N* $K_2Cr_2O_7$, using a Roberts electrometric titration apparatus[6] or equivalent to determine the potential. The volume of standard $K_2Cr_2O_7$ necessary to effect oxidation of the ferrous iron is the volume required in passing from the first point of inflection as shown by the potentiometer reading, which indicates the oxidation of residual $SnCl_2$ and initial oxidation of the ferrous iron, to the second point of inflection, which indicates the complete transformation of the iron to the ferric condition. The points of inflection are best determined by means of a graph in which the volumes of $K_2Cr_2O_7$ used are plotted against the resulting electromotive force.

[5] The quantity of H_2SO_4 specified here will be sufficient for sands containing as much as 0.5 per cent total bases (oxides of Fe, Al, Ti, Zr, Ca, Mg, Na, K, etc.). For larger amounts of the bases, proportionately larger quantities of H_2SO_4, up to 20 ml. for 10 per cent Al_2O_3, must be used.

[6] J. C. Hostetter and H. S. Roberts, "Electrometric Titrations, with Special Reference to the Determination of Ferrous and Ferric Iron," *Journal*, Am. Chemical Soc., Vol. 41, p. 1337 (1920); "Electrometric Determination of Iron," *Journal*, Am. Ceramic Soc., Vol. 10, April, 1927 (p. 100 of the *Bulletin*).

Solution for Determination of Al_2O_3, TiO_2, ZrO_2, CaO, and MgO (P_2O_5, V_2O_5)

7. Transfer 25.00 g. of the sand, dried at 105 to 110 C., to a 250 to 300-ml. platinum dish. Thoroughly moisten the sample with 20 ml. of diluted H_2SO_4 (1:1) and add slowly, while stirring constantly with a platinum rod or spatula, 200 ml. of HF. Evaporate to fumes of H_2SO_4 and add a few drops of HNO_3 to the hot solution to destroy organic matter. Repeat the hydrofluoric acid treatment, using 50 ml. of HF. Cool the dish and contents, rinse the walls of the dish with water, and repeat the evaporation to fumes of H_2SO_4. When cool, add 150 ml. of water and heat until soluble salts have dissolved. Filter, wash the residue and paper with hot water, and reserve the filtrate. Ignite the insoluble residue, fuse with a small amount of Na_2CO_3, and dissolve the melt in diluted H_2SO_4 (1:5). Filter, wash the residue and paper, and combine the filtrate with that previously reserved. Ignite the filter and insoluble residue. Fuse with a minimum quantity of potassium pyrosulfate ($K_2S_2O_7$), dissolve the melt in diluted H_2SO_4 (1:5), and combine with the reserved filtrates. Dilute to 250 ml. and saturate the solution with H_2S. Digest, preferably overnight, filter, wash with diluted H_2SO_4 (1:99) saturated with H_2S, and discard the sulfide precipitate. Boil the filtrate to expel H_2S, oxidize with bromine water, boil off the excess of bromine, and precipitate with NH_4OH, using methyl red as the indicator. Filter, and wash the paper and precipitate thoroughly with a hot neutral solution of NH_4NO_3 (2 per cent). Acidify, and reserve the filtrate for the determination of calcium and magnesium (see Sections 12 to 14). Transfer the paper and precipitate to a 400-ml. beaker and add 20 ml. of HNO_3 and 10 ml. of H_2SO_4. Cover, heat cautiously until the solution begins to blacken, and then add HNO_3 drop by drop, allowing each drop to flow down the beaker wall, until all carbonaceous matter has been destroyed. Heat to strong fumes of H_2SO_4, cool, rinse the cover glass and beaker walls, and again heat to fumes of H_2SO_4. Cool, add 150 ml. of water, and heat to dissolve any anhydrous sulfates. Cool the solution, transfer to a 200-ml. volumetric flask, and dilute to the mark. Mix thoroughly, and divide the solution into two 100-ml. aliquot portions.

Determination of Al_2O_3, Fe_2O_3, TiO_2, and ZrO_2 (P_2O_5, V_2O_5)

8. Dilute one of the 100-ml. aliquot portions (Section 7) to 250 ml., heat to boiling, and precipitate with NH_4OH, using methyl red as the indicator. Filter, and wash paper and precipitate thoroughly with a hot, neutral solution of NH_4NO_3 (2 per cent). Ignite the paper and precipitate in a platinum crucible weighed with cover. Moisten the ignited residue with water, add 5 to 10 drops of diluted H_2SO_4 (1:1) and 5 ml. of HF. Evaporate to expel the excess of acids, and gradually increase the temperature until all H_2SO_4 has been expelled and the sulfates decomposed. Cover the crucible and ignite at approximately 1100 C. Cool over a good desiccant, and weigh. Repeat the ignition until constant weight is obtained. The weight of the oxides multiplied by 8 represents the combined percentages of Al_2O_3, Fe_2O_3, TiO_2, and ZrO_2 (P_2O_5, V_2O_5).

Determination of Fe_2O_3, TiO_2, and ZrO_2 (V_2O_5)

9. Dilute the other 100-ml. aliquot portion (Section 7) to 200 ml., add 15 ml. of H_2SO_4, and cool the solution to 10 C. Add slowly, while stirring con-

stantly, a cold solution of cupferron (6 per cent) until no further precipitation occurs. Stir in a little macerated paper, allow the precipitate to settle, filter, and wash the paper and precipitate thoroughly with cold diluted H_2SO_4 (1:9) containing 1.5 g. of cupferron per liter. Drain the paper and precipitate, and transfer to a platinum crucible weighed with cover. Dry the precipitate cautiously and ignite slowly until all carbonaceous matter is destroyed. Cool the crucible, and treat with H_2SO_4 and HF to remove any SiO_2. Evaporate to dryness and then ignite at approximately 1100 C. Cool over a good desiccant, and weigh. Repeat the ignition until constant weight is obtained. The weight of the oxides multiplied by 8 represents the combined percentages of Fe_2O_3, TiO_2, and ZrO_2 (V_2O_5).

Determination of Al_2O_3 (P_2O_5)

10. The percentage of oxides obtained from the ammonium hydroxide precipitation of the first aliquot portion minus the percentage of oxides obtained from the cupferron precipitation of the second aliquot portion represents the percentage of Al_2O_3 (P_2O_5).

Determination of TiO_2 and ZrO_2

11. The percentage of oxides obtained from the cupferron precipitation, minus the percentage of Fe_2O_3 as determined on the separate sample, represents the combined percentage of TiO_2 and ZrO_2 (V_2O_5). If desired, TiO_2 and ZrO_2 may be determined by fusing the cupferron precipitate with $K_2S_2O_7$, taking up the melt in diluted H_2SO_4 (1:9), and first determining titanium colorimetrically with H_2O_2 and then determining zirconium by precipitation as phosphate. If V_2O_5 is present, it imparts a reddish-brown color in the titanium test that is not dissipated by a drop or two of HF.

Determination of CaO

12. Evaporate to 100 ml. the reserved filtrate obtained in the NH_4OH precipitation in Section 7, and add two drops of methyl red (0.02 per cent) and 1 g. of oxalic acid. Heat the solution to boiling, and slowly add NH_4OH until the solution is alkaline. Digest, cool, filter on a small S. & S. No. 589 blue band, or equivalent, paper, and wash the precipitate and paper with a cool, neutral solution of ammonium oxalate (0.1 per cent). Acidify, and reserve the filtrate and washings. Transfer the precipitate and paper to a platinum crucible (weighed with cover), and ignite. Moisten the oxide with a few drops of water, add 10 drops of diluted H_2SO_4 (1:9), and evaporate to dryness. Heat the crucible and contents to dull redness, cool, and weigh. Repeat the ignition until constant weight is obtained. The weight of the $CaSO_4$ multiplied by 1.6476 represents the percentage of CaO.

Determination of MgO

13. Evaporate to 100 ml. the filtrate from the ammonium oxalate precipitation (Section 12). Cool the solution, and add 1 g. of $(NH_4)_2HPO_4$. Stir the solution vigorously, slowly add NH_4OH until the solution is alkaline, and then add 10 ml. of NH_4OH. Allow the solution to stand overnight. Filter, and wash with diluted NH_4OH (5:95). Dry the precipitate and paper in a platinum crucible (weighed with cover), char the paper slowly, and burn off the carbon by gradually increasing the temperature. Cover the crucible, and ignite at 1000 to 1100 C. for 30 min. Cool in a desiccator, and weigh. Repeat the ignition to constant weight. The weight of the $Mg_2P_2O_7$ multiplied by 1.4484 represents the percentage of MgO.

Determination of CaO and MgO (Alternate)

14. If desired, the calcium and magnesium may be precipitated in the evaporated filtrate (Section 12) as follows: Add from 1 to 3 g. of $(NH_4)_2HPO_4$ dissolved in a few milliliters of water, and render the solution ammoniacal. Stir thoroughly to induce precipitation, add 10 ml. of NH_4OH, and allow to stand for 4 to 6 hr. Filter, and wash the precipitated phosphates with cool, diluted NH_4OH (5:95). Ignite the precipitate in a platinum crucible (weighed with cover), cool, and weigh as $Ca_3(PO_4)_2$ plus $Mg_2P_2O_7$. Transfer the weighed residue to a small beaker and dissolve in a little diluted H_2SO_4 (1:9), avoiding more than a 0.5 ml. excess. Add 25 ml. of 75 per cent ethyl alcohol and allow the solution to stand several hours, or overnight if the amount of calcium is very small. Filter, and wash the precipitate with 75 per cent ethyl alcohol. Dissolve the dry precipitate in a very little diluted HCl (1:9) and precipitate as oxalate in as small a volume of solution as possible. Filter, wash, ignite, and weigh the calcium as CaO. The magnesium is determined, by difference, by subtracting the weight of the $Ca_3(PO_4)_2$, as calculated from the weight of the CaO found, from the weight of the combined phosphates, and calculating the remainder to MgO.

Standard Methods of

CHEMICAL ANALYSIS OF SODA-LIME GLASS[1]

A.S.T.M. Designation: C 169 – 43

ADOPTED, 1943.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 169; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. (*a*) These methods for the chemical analysis of glass cover procedures for use in the referee analysis and in the routine analysis of soda-lime glasses containing the usual constituents of such glasses and not more than 0.5 per cent of fluorine, 2.0 per cent of barium oxide (BaO), 2.0 per cent of boric oxide (B_2O_3), 0.10 per cent of phosphorus pentoxide (P_2O_5), 0.10 per cent of zinc oxide (ZnO), 0.05 per cent of antimony oxide (Sb_2O_3), and 0.05 per cent of lead oxide (PbO). These methods do not preclude the use of other methods that yield results within permissible variations. In any case, the analyst should check the procedure and technique that are employed by means of a National Bureau of Standards standard sample having a composition comparable with that of the material under test. A list of these standard samples is given in the Bureau's Supplement to *Circular C398*.

(*b*) The analytical procedures appear in the following order:

	Sections
Procedures for Referee Analysis:	
Silicon Dioxide	6
Ferric Oxide	7 and 8
Determination of BaO, $Al_2O_3 + P_2O_5$, CaO, and MgO	9 to 12
Sodium Oxide and Potassium Oxide	13 to 17
Boric Oxide	18 to 20
Sulfur Trioxide	21 and 22
Procedures for Routine Analysis:	
Silicon Dioxide	23
Determination of BaO, R_2O_3, CaO, MgO, Na_2O, and K_2O	24 to 31
Boric Oxide	32 and 33
Fluorine	34 and 35
Sulfur Trioxide	36

Reagents

2. (*a*) *Purity of Reagents.*—All reagents shall be free of such amounts of impurities as would endanger the test in question. Reagents conforming to the specifications of the Committee on Analytical Reagents of the American Chemical Society are recommended. In all cases, blank determinations on all reagents used shall be carried through each step of the procedure and the final result corrected as may be indicated. The term "water" as used in these methods refers only to distilled water.

(*b*) *Concentration of Acids and Ammonium Hydroxide.*—Unless otherwise stated, the concentrated reagents, as furnished by the manufacturer, are to be used. For example, hydrochloric acid or HCl refers to acid of specific

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee C-14 on Glass and Glass Products.

[2] Prior to adoption as standard, these methods were published as tentative from 1941 to 1943, being revised in 1943.

gravity 1.19, sulfuric acid or H_2SO_4 refers to acid of specific gravity 1.84, and ammonium hydroxide or NH_4OH refers to the reagent of specific gravity 0.90. Diluted acids and ammonium hydroxide are designated as follows: diluted HCl (5:95) means 5 volumes of the concentrated acid of specific gravity 1.19 diluted with 95 volumes of water, and diluted NH_4OH (5:95) means 5 volumes of concentrated ammonium hydroxide of specific gravity 0.90 diluted with 95 volumes of water.

Filter Papers

3. Throughout these methods, filter papers will be designated as "loose" or "tight," without naming brands or manufacturers. "Loose papers" will mean the type of ashless papers that are generally used by analysts for filtering aluminum hydroxide or large amounts of silica. "Tight papers" will mean such ashless papers as are generally used by analysts for filtering barium sulfate.

Preparation of Sample for Analysis

4. The sample for analysis shall pass a No. 60 (250-micron) sieve and shall be dried at 105 to 110 C. It should be noted that soda-lime glasses may absorb and fix carbon dioxide and water during the grinding process and in storage. If the composition of the unground glass is desired, determinations of the loss on ignition at 1000 C. shall be made on an appropriate piece of the unground glass and on the powdered sample. From the two results obtained, the amount of carbon dioxide and water absorbed by the powdered glass can be calculated and the proper correction shall be made.

Probable Accuracy of Results

5. (*a*) The probable accuracy of results that can be expected by the use of the procedures described in Sections 6 to 36 is shown in the following tabulation:

Constituent	Probable Accuracy of Results, per cent: Referee Analysis	Routine Analysis
SiO_2	±0.1	±0.1 in absence of B_2O_3; +0.1 to +0.2 in presence of B_2O_3
Fe_2O_3	±0.005	..
BaO	−0.02	±0.05
$Al_2O_3 + P_2O_5$	±0.05	..
CaO	±0.05	±0.05
MgO	±0.05	±0.05
Na_2O	±0.2	±0.2
K_2O	±0.03	±0.05
B_2O_3	±0.05	±0.05
SO_3	±0.03	±0.05
"R_2O_3"	...	±0.05 (Al_2O_3 less than 1 per cent) to ±0.1 (Al_2O_3 more than 1 per cent)

(*b*) It is recommended that *reported* results be rounded off as follows:

Percentage	Number of Significant Figures Retained After Rounding Off
1 to 100	3
0.1 to 0.9	2
0.01 to 0.09	1 or 2
Under 0.01	1

(*c*) *Recorded* results should be carried to one more significant figure than required in Paragraph (*b*).

PROCEDURES FOR REFEREE ANALYSIS

SILICON DIOXIDE

Procedure

6. (*a*) Transfer 0.5000 g. of the prepared sample (Section 4) and 0.50 g. of Na_2CO_3 to a 75-ml. platinum dish,[3] and intimately mix the contents by means of the flattened end of a glass rod. Brush the mixture into the center of the dish, flatten the charge so that it covers a space about 3 cm. in diameter, and then cover it as evenly as possible with an additional 0.50 g. of Na_2CO_3. Heat gradually at first (preferably over a low flame) and finally at the temperature

[3] Dishes such as are used in the determination of alkalies are satisfactory, but special long-lip 75-ml. dishes of platinum alloyed with 3.5 per cent of rhodium are preferable.

required to give a clear melt. Maintain at this temperature for 10 min. Remove the dish from the source of heat, and cover it to prevent loss of small pieces of the solidified melt that might be ejected during cooling. A platinum cover may be used during the fusion, but this is not necessary, since no spattering occurs.

(*b*) Add 20 ml. of diluted HCl (1:1) to the cooled melt, cover the dish, and digest on the steam bath until disintegration is complete. If desired, the cooled melt may be digested in water before the acid is added. The action of the acid can be hastened by occasionally crushing the layer of insoluble matter that tends to cover the unattacked portion of the fused mass. As soon as disintegration is complete, crush all flakes of solid matter with the flattened end of a glass rod. Evaporate to dryness on the steam bath, cool, drench the dry residue with 5 ml. of HCl, and then add 15 ml. of warm water. Digest on the steam bath for a few minutes, and filter through a loose 9-cm. filter paper. Wash the dish, residue, and filter paper with ten 3-ml. portions of hot diluted HCl (2:98), but make no attempt to transfer all of the residue from the dish to the paper. Place the filter containing the residue in the platinum dish in which the fusion and dehydration were made, and evaporate the filtrate to dryness on the steam bath in another dish, preferably of platinum. Heat the residue at 105 to 110 C. for 15 min. Cool, drench the dry residue with HCl, dilute, filter through a small, tight filter paper, and wash as before. Transfer all the residue obtained in the second dehydration to the paper, and then place this paper with the paper containing the first residue in the platinum dish in which the fusion was made.

(*c*) Ignite the contents of the dish slowly at first and finally at approximately 600 C. until the carbon is gone. Add 3 or 4 drops of diluted H_2SO_4 (1:1) if a high nonsilica residue is expected, and ignite slowly at first and finally for 30 min. to 1 hr. at 1200 C. Cool in a desiccator, and weigh. Repeat ignition to constant weight. Treat the ignited residue with a few drops of diluted H_2SO_4 (1:1) and about 10 ml. of HF. Evaporate the volatile constituents on the steam bath, drive off the H_2SO_4 by heating over a free flame, and finally ignite the residue for 1 to 2 min. at 1200 C. The difference between the first and second weights, with a proper correction for a blank for the reagents, represents the weight of SiO_2 in the sample.

(*d*) *Calculation.*—Calculate the percentage of SiO_2 as follows:

$$SiO_2, \text{ per cent} = \text{wt. of } SiO_2 \times 200$$

NOTE.—The small amounts of fluorine that may be present in these glasses may cause a small but negligible negative error in the results for SiO_2, and the B_2O_3 may cause a small but negligible positive error.

FERRIC OXIDE

Reagents

7. (*a*) *Potassium Dichromate (0.005 N).*—Recrystallize, three times, the purest grade of $K_2Cr_2O_7$ obtainable. Dry the crystals at 150 C., grind to a fine powder, and dry at 200 C. until constant weight is attained. Dissolve 2.4516 g. of the recrystallized salt in water and dilute to exactly 10 liters. One milliliter of exactly 0.005 *N* $K_2Cr_2O_7$ represents 0.000399 g. of Fe_2O_3.

(*b*) *Stannous Chloride Solution.*—Dissolve 150 g. of $SnCl_2 \cdot 2H_2O$ in 100 ml. of HCl and dilute to 1000 ml. with water. Keep a few pieces of metallic tin in the bottle.

Procedure

8. Transfer 10 g. (Note) of the prepared sample (Section 4) to a 200-ml.

platinum dish. Moisten the sample with water, and add 50 ml. of diluted H_2SO_4 (1:1). Add cautiously 100 ml. of HF, mix thoroughly by stirring with a platinum rod or spatula, and evaporate to about 30 ml. on a steam or sand bath. Add 50 ml. of HF and evaporate to fumes of H_2SO_4. To the gently fuming solution, add cautiously, drop by drop, a few drops of HNO_3 to destroy organic matter. Cool, rinse the walls of the dish with water, and again heat to strong fumes of H_2SO_4 in order to expel all HNO_3. Cool the dish and contents, and add 100 ml. of water. Heat until soluble salts are in solution, and add strips of iron-free magnesium ribbon to precipitate platinum introduced during the decomposition of the sample. The treatment with magnesium may be done conveniently by wrapping the ribbon around the end of the glass stirring rod. The addition of sufficient magnesium is indicated when the ferric iron has been completely decolorized. After all the magnesium has been dissolved, boil for a few minutes, filter if platinum has separated, and wash the paper with hot water. Adjust the volume to 50 ml. Oxidize any reducing substances that may be present by adding 0.1 *N* $KMnO_4$ until a permanent pink coloration is produced. Add 50 ml. of HCl, heat to boiling, and reduce the iron with the minimum amount of a solution of $SnCl_2$. Titrate electrometrically with standard 0.005 *N* $K_2Cr_2O_7$, using a Roberts electrometric titration apparatus[4] or equivalent to determine the potential. The volume of standard $K_2Cr_2O_7$ necessary to effect oxidation of the ferrous iron is the volume required in passing from the first point of inflection as shown by the potentiometer reading, which indicates the oxidation of residual $SnCl_2$ and initial oxidation of the ferrous iron, to the second point of inflection, which indicates the complete transformation of the iron to the ferric condition. The points of inflection are best determined by means of a graph in which the volumes of $K_2Cr_2O_7$ used are plotted against the resulting electromotive force.

NOTE.—This procedure is satisfactory for determining iron up to a total of 10 mg. of Fe_2O_3. For larger quantities, the size of the sample should be so reduced that the 10 mg. is not exceeded. This may be accomplished by using a smaller sample of the glass, or preferably, by taking an appropriate aliquot portion of the solution obtained after decomposition of the 10-g. sample.

DETERMINATION OF BaO, Al_2O_3 + P_2O_5, CaO, AND MgO

Preparation of the Solution and Determination of BaO

9. (*a*) Transfer 2.50 g. of the prepared sample (Section 4) to a 250- to 300-ml. platinum dish. Thoroughly moisten the sample with 20 ml. of diluted H_2SO_4 (1:1) and add slowly, while stirring constantly with a platinum rod or spatula, about 30 ml. of HF. Evaporate to fumes of H_2SO_4, and add a few drops of HNO_3 to the hot solution to destroy organic matter. Repeat the hydrofluoric acid treatment, using 20 ml. of HF. When the acid is fuming strongly, cool the dish and contents, rinse the walls of the dish with water, repeat the evaporation to fumes of H_2SO_4, and continue the fuming for 10 min. Cool, add 250 ml. of diluted HCl (1:99), and heat until soluble salts have dissolved. Cool, and allow to stand 2 hr. or longer. Filter through a tight paper, wash the residue and paper with hot water, and reserve the filtrate and washings. Ignite the insoluble residue in platinum, fuse with 3 g. or less of Na_2CO_3, and leach the melt with hot water. Filter through a loose paper, wash with hot water, and reserve

[4] J. C. Hostetter and H. S. Roberts, "Electrometric Titrations, with Special Reference to the Determination of Ferrous and Ferric Iron," *Journal*, Am. Chemical Soc., Vol. 41, p. 1337 (1920); "Electrometric Determination of Iron," *Journal*, Am. Ceramic Soc., Vol. 10, April, 1927 (p. 100 of the *Bulletin*).

the filtrate and washings. Dissolve the insoluble matter in the crucible, in the beaker, and on the filter paper in 25 ml. of hot diluted HCl (5:95). Wash the beaker, crucible, and filter thoroughly with hot water, dilute the solution to 200 ml., heat to boiling, and add 4 ml. of diluted H_2SO_4 (1:1). Digest on the steam bath for about 1 hr., cool, and allow to stand 2 hr. or longer. Filter through a tight paper, and wash thoroughly with hot water. Reserve the filtrate and washings. Ignite the precipitate slowly in a porcelain or platinum crucible until the carbon is burned; then heat at about 1000 C. for 15 min. Cool in a desiccator and weigh.

(*b*) *Calculation.*—Calculate the percentage of BaO as follows:

$$\text{BaO, per cent} = \text{wt. of } BaSO_4 \times 26.28$$

Determination of $Al_2O_3 + P_2O_5$

10. (*a*) Combine the three reserved filtrates (Section 9 (*a*)) (from the first precipitation of $BaSO_4$, from the leaching of the sodium carbonate fusion, and from the second precipitation of $BaSO_4$). Neutralize the excess acid with NH_4OH and then add 3 ml. of HCl. Adjust the volume to 300 ml. and saturate the solution with H_2S. Digest, preferably overnight, filter through a tight paper, wash with diluted H_2SO_4 (1:99) saturated with H_2S, and discard the sulfide precipitate. Boil the filtrate to expel H_2S, oxidize with bromine water, boil off the excess of bromine, and precipitate with NH_4OH, using methyl red as the indicator. Filter, and allow the solution to drain. Reserve the filtrate. Transfer the paper and precipitate to the beaker, and add 10 ml. of diluted HCl (1:1). Stir the paper to a pulp, warm until the hydroxides are dissolved, and add 150 ml. of water. Heat the solution to boiling, and precipitate with NH_4OH as before. Filter, and wash the paper and precipitate thoroughly with a hot neutral solution of NH_4Cl (2 per cent). Combine the two filtrates, acidify, and reserve them for the determination of calcium and magnesium (Sections 11 and 12). Transfer the paper and precipitate to a 400-ml. beaker, and add 20 ml. of HNO_3 and 10 ml. of H_2SO_4. Cover, heat cautiously until the solution begins to blacken, and then add HNO_3 drop by drop, allowing each drop to flow down the beaker wall, until all carbonaceous matter has been destroyed. Heat to strong fumes of H_2SO_4, cool, rinse the cover glass and beaker walls, and again heat to fumes of H_2SO_4. Cool, add 150 ml. of water, and heat to dissolve any anhydrous sulfates. Cool the solution, transfer to a 200-ml. volumetric flask, and dilute to the mark. Mix thoroughly, and divide the solution into two 100-ml. aliquot portions.

(*b*) *Determination of Al_2O_3, P_2O_5, Fe_2O_3, TiO_2, ZrO_2, and V_2O_5 (If the glass contains chromium, Cr_2O_3 will be included here).*—Dilute one of the 100-ml. aliquot portions to 250 ml., heat to boiling, and precipitate with NH_4OH, using methyl red as the indicator. Filter, and wash paper and precipitate thoroughly with a hot, neutral solution of NH_4NO_3 (2 per cent). Discard the filtrate. Ignite the paper and precipitate in a platinum crucible weighed with cover. Moisten the ignited residue with water, then add 5 to 10 drops of diluted H_2SO_4 (1:1) and 5 ml. of HF. Evaporate to expel the excess of acids, and gradually increase the temperature until all H_2SO_4 has been expelled and the sulfates decomposed. Cover the crucible and ignite at approximately 1100 C. Cool over a good desiccant, and weigh. Repeat the ignition until constant weight is obtained. The weight of the oxides multiplied by 80 represents the combined percentages of Al_2O_3, P_2O_5, Fe_2O_3, TiO_2, ZrO_2, V_2O_5, and Cr_2O_3.

(*c*) *Determination of Fe_2O_3, TiO_2, ZrO_2, and V_2O_5.*—Dilute the other 100-ml.

aliquot portion (Paragraph (*a*)) to 200 ml., add 15 ml. of H_2SO_4, and cool the solution to 10 C. Add slowly, while stirring constantly, a cold solution of cupferron (6 per cent) until no further precipitation occurs. (Prepare the cupferron solution by dissolving 6 g. of cupferron reagent in cold water immediately before use. Filter, and use the clear solution.) Stir in a little macerated filter paper, allow the precipitate to settle, filter through a tight paper, and wash the paper and precipitate thoroughly with cold diluted H_2SO_4 (1:9) containing 1.5 g. of cupferron per liter. Drain the paper and precipitate, and transfer to a platinum crucible weighed with cover. Dry the precipitate cautiously at about 60 C., and ignite slowly until all carbonaceous matter is destroyed. Cool the crucible, and treat with H_2SO_4 and HF to remove any SiO_2. Evaporate to dryness and then ignite at approximately 1100 C. Cool over a good desiccant, and weigh. Repeat the ignition until constant weight is obtained. The weight of the oxides multiplied by 80 represents the combined percentages of Fe_2O_3, TiO_2, ZrO_2, and V_2O_5.

(*d*) *Determination of* $Al_2O_3 + P_2O_5$.—The percentage of oxides obtained from the ammonium hydroxide precipitation of the first aliquot portion (Paragraph (*b*)) minus the percentage of oxides obtained from the cupferron precipitation of the second aliquot portion (Paragraph (*c*)) represents the percentage of $Al_2O_3 + P_2O_5$. Chromium is not precipitated by cupferron and will be counted with the $Al_2O_3 + P_2O_5$ if the glass contains chromium.

If the glass contains P_2O_5, some of it will be found in the ignited oxides obtained in accordance with Paragraph (*b*). To test for P_2O_5, fuse the ignited oxides with Na_2CO_3, dissolve the cooled melt in diluted HNO_3, and add molybdate reagent. If a precipitate of phosphomolybdate appears, filter, determine the P_2O_5 by the customary alkalimolybdate method, and deduct from the weight of the ignited $Al_2O_3 + P_2O_5$.

Determination of CaO

11. (*a*) Evaporate to 150 ml. the acidified and combined reserved filtrates obtained in the NH_4OH precipitations (Section 10(*a*)) and transfer to a 200-ml. volumetric flask. Dilute to the mark and mix thoroughly. To a 100-ml. aliquot portion add two drops of methyl red (0.02 per cent) and 1 g. of oxalic acid. Heat the solution to boiling, and slowly add NH_4OH until the solution is alkaline. Digest for 1 hr. on the steam bath, cool to room temperature, filter on a small, tight paper, and wash the precipitate and paper two or three times with a cool, neutral solution of ammonium oxalate (0.1 per cent). Reserve the filtrate and washings. Return the paper and precipitate to the precipitating vessel, add 10 ml. of diluted HCl (1:1), and stir the paper to a pulp. Dilute the solution to 200 ml., and add 0.2 g. of oxalic acid. Heat the solution to boiling, and slowly add NH_4OH until the solution is alkaline. Digest, filter, and wash as before. Combine the filtrate and washings with the reserved filtrate and washings, and acidify with HCl. Transfer the paper and precipitate to a platinum crucible (weighed with cover), and heat until carbon has been destroyed. Cover tightly, ignite for about 5 min. at 1200 C., cool over a good desiccant, and weigh as CaO. The crucible should be reheated and the weight checked because of possible absorption of moisture and CO_2 during the first weighing.

(*b*) *Calculation.*—Calculate the percentage of CaO as follows:

$$\text{CaO, per cent} = \text{wt. of CaO} \times 80$$

Determination of MgO

12. (*a*) Evaporate to 150 to 200 ml. the filtrates from the ammonium oxalate precipitations (Section 11(*a*)). Cool the solution, and add 1 g. of $(NH_4)_2HPO_4$. Stir the solution vigorously, slowly add NH_4OH until the solution is alkaline, and then add 10 ml. of NH_4OH. Allow the solution to stand 4 hr. or overnight. Filter through a tight paper, wash the beaker, paper, and precipitate moderately with diluted NH_4OH (5:95), and discard the filtrate. Place the beaker under the funnel, and dissolve the precipitate in 25 to 50 ml. of hot, diluted HCl (1:9). Add 0.1 g. of $(NH_4)_2HPO_4$, dilute to 75 ml., cool, and then add NH_4OH drop by drop, while stirring constantly, until the solution is alkaline. Finally add 5 ml. in excess, and let stand 4 hr. or overnight. Filter through a tight paper, transfer all the precipitate to the paper, and wash with cool diluted NH_4OH (5:95). Dry the precipitate and paper in a platinum crucible (weighed with cover), char the paper slowly, and burn off the carbon by gradually increasing the temperature. Cover the crucible, and ignite at 1000 to 1100 C. for 30 min. Cool in a desiccator, and weigh. Repeat the ignition to constant weight.

(*b*) *Calculation.*—Calculate the percentage of MgO as follows:

$$\text{MgO, per cent} = \text{wt. of } Mg_2P_2O_7 \times 29.0$$

SODIUM OXIDE AND POTASSIUM OXIDE

Reagents

13. (*a*) *Ammonium Chloride.*—The reagent grade of NH_4Cl is satisfactory if a test shows that 1 g. yields not more than 0.1 mg. of nonvolatile residue when the NH_4Cl is volatilized by heating and the residue is ignited at 700 to 800 C. If the reagent grade is not satisfactory, sublime it slowly, crush the sublimed material in an agate mortar, and store the fine powder in a well-stoppered bottle.

(*b*) *Calcium Carbonate.*—The reagent grade of $CaCO_3$ is usually unsatisfactory. Dissolve the purest $CaCO_3$ obtainable in diluted HCl (1:3), precipitate with NH_4OH and $(NH_4)_2CO_3$, wash thoroughly with hot water, dry at about 105 C., and store in a well-stoppered bottle.

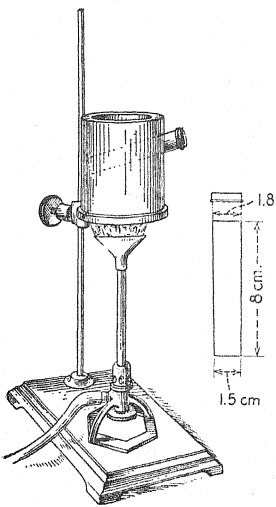


FIG. 1.—Apparatus for Determining Sodium Oxide and Potassium Oxide.

(*c*) *Chloroplatinic Acid Solution.*—Dissolve 15 g. of chloroplatinic acid ($H_2PtCl_6 \cdot 6H_2O$) in 50 ml. of water containing 5 ml. of diluted HCl (1:1). One milliliter will contain about 0.1 g. of platinum; 1.31 ml. of such a solution is required for 0.1 g. of KCl, and 1.68 ml. for 0.1 g. of NaCl.

Apparatus

14. *Platinum Crucible.*—A platinum crucible of suitable size for treating 0.5 g. of sample is 8 cm. in length, 1.8 cm. in diameter at the mouth, and 1.5 cm. in diameter at the bottom, and has a weight of about 25 g. The crucible and the chimney to hold it are shown in Fig. 1.

Determination of NaCl and KCl

15. (*a*) Transfer 0.5000 g. of the prepared sample (Section 4) and 0.50 g. of NH_4Cl to a large, smooth agate mortar placed on a piece of glazed paper. Grind with a pestle until the sample and the NH_4Cl are thoroughly mixed. Next add, in several portions, nearly all of 4 g. of $CaCO_3$, and mix very thoroughly in the mortar. Line the bottom of the crucible with a little of the $CaCO_3$, and transfer the mixture to the crucible by the aid of a spatula having a blade that is somewhat flexible and not too broad. Use the rest of the $CaCO_3$ for rinsing the mortar and pestle, and transfer the rinsings also. All this should be done over the piece of glazed paper.

(*b*) Cap the crucible, tap it gently to make the powder settle, and place it in an inclined position in a clay cylinder (Fig. 1) or through a hole in a piece of stout asbestos board clamped at a slight angle. Heat the part within the cylinder or projecting through the board by a fishtail flame placed considerably beneath the crucible. Continue gentle heating for about 15 min., or until the odor of ammonia is no longer perceptible. Do not heat so that NH_4Cl escapes. Replace the flame by one or two burners of any type that will give the proper temperature, and heat at about 1000 C. for 30 to 60 min.

(*c*) Cool, transfer the sintered cake to a 12-cm. porcelain dish, pour hot water into the crucible, and digest until all remaining matter can be washed out or until it is thoroughly extracted. In most instances, the entire sintered cake can be loosened from the crucible by lightly tapping. Slake the cake in the dish with 1 to 2 ml. of water at first, then add 50 ml. more, until thoroughly disintegrated. If the cake clings to the crucible, the slaking must be done without removing it. When the cake has disintegrated and the solution is saturated with $Ca(OH)_2$, filter by decantation upon a 9-cm. filter and let the filtrate run into a capacious dish, preferably of platinum. Keep as much as possible of the residue in the porcelain dish. Add 50 ml. more of hot water, break up any lumps by gentle pressure with a pestle, let settle, and again decant. Repeat the extraction at least three more times. Wash the residue in the paper and dish with a saturated solution of $Ca(OH)_2$ free of sodium and potassium. The final residue, which is to be discarded, should dissolve completely in HCl and will contain very little, if any, alkalies if the work has been carefully done.

(*d*) To the filtrate and washings, add a few drops of NH_4OH and then an excess of solid $(NH_4)_2CO_3$. The presence of an excess can be determined by allowing the precipitate of $CaCO_3$ to settle and then adding small pieces of $(NH_4)_2CO_3$ and noting whether further precipitation takes place. Heat until effervescence ceases, and boil carefully for at least 3 min. to expel CO_2. Rinse and remove the cover, filter into a 1-liter casserole, and wash the dish, paper, and residue with five 10-ml. portions of hot water. Reserve the filtrate. Dissolve the precipitate in as little diluted HCl (1:4) as possible, and reprecipitate with $(NH_4)_2CO_3$ as before. Filter, and add the filtrate to the reserved filtrate.

(*e*) Evaporate the combined filtrates to dryness, and heat gently to remove ammonium salts. Dissolve the residue in 25 ml. of water, add a few drops of NH_4OH, heat to boiling, add $(NH_4)_2C_2O_4 \cdot H_2O$ (saturated solution) until an excess of about 5 drops is present, and let stand for 2 hr. or preferably overnight.[5] Filter through a tight paper to remove the calcium oxalate, catch the filtrate in a 100-ml. platinum dish, and wash paper and residue with a cold 0.1 per cent solution of $(NH_4)_2C_2O_4 \cdot H_2O$.

(*f*) Evaporate the filtrate and washings to dryness, cover the dish, and heat at 110 to 120 C. Cautiously ignite, with cover on at first, until ammonium salts have been volatilized. Cool, rinse, and remove the cover glass, and dissolve the residue in a little water. Add a few drops of HCl to decompose any alkali carbonate that may have been formed by action of the ammonium oxalate during the ignition. Again evaporate to dryness. Add 5 ml. of HCl and 5 ml. of methyl alcohol. Cautiously evaporate to dryness to drive off any boron that may be present. Cover the dish, heat in an oven at 135 to 140 C., and ignite very gently (until salts just begin to melt) to constant weight.

(*g*) Dissolve the salts in hot water, filter through a small ashless filter paper, and wash with hot water. Ignite the paper in the dish, cool, weigh, and subtract the second weight from the first in order to obtain the weight of NaCl + KCl.

Determination of K_2O

16. (*a*) The following procedure shall be carried out in an atmosphere free of ammonia and the fumes of ammonium salts: Dissolve the mixed chlorides (Section 15) in a little water, transfer to a porcelain dish, and add enough chloroplatinic acid to react with all of both the sodium and the potassium. Evaporate on the steam bath until the solution is syrupy but solidifies on cooling. Do not evaporate to dryness. Cool, drench the residue with ethyl alcohol (80 per cent by volume), and filter by decantation through a small paper. Wash by decantation with more of the alcohol, crushing the crystals with a small pestle or the widened and rounded end of a short glass rod. Dry the dish and paper to remove adhering alcohol completely.

(*b*) Dissolve the precipitate on the paper in hot water, catching the solution in a weighed platinum crucible or small platinum dish. Evaporate to dryness, and add the salt that is still in the porcelain dish. If the residue is large and lumpy, redissolve in water, and again evaporate to dryness. Cover and heat for 1 hr. at 130 C. in an oven (100 C. suffices for very small amounts of fine-grain precipitate). Cool, and weigh as K_2PtCl_6. Multiply by 0.194 to obtain the weight of K_2O and by 0.307 to obtain the corresponding weight of KCl.

Determination of Na_2O

17. Calculate the weight of sodium oxide by subtracting the weight of KCl obtained in Section 16 from the weight of NaCl + KCl, and multiplying the remainder by 0.530.

BORIC OXIDE

Apparatus

18. The apparatus, shown in Fig. 2, shall consist of the following: *A*, a U-tube containing mercury, used as a safety trap; *B*, a 500-ml. round-bottom distilling flask for methyl alcohol, containing a capillary "boiling tube" of

[5] If the material contains sulfates, these must be removed by adding a slight excess of $BaCl_2$ solution (10 per cent) to the water solution of the residue, heating to boiling, letting settle, and filtering. In this case, the solution must be treated with NH_4OH and $(NH_4)_2CO_3$ and filtered in order to remove the excess of barium before the ammonium oxalate is added.

3-mm. bore, the bore being sealed 10 mm. from the lower end; *C*, a 300-ml. round-bottom flask; *D*, a No. 4 (360-ml.) casserole; *E*, a glass condenser; *F*, a 200-ml. Erlenmeyer flask; and *G*, a U-tube containing water to prevent the escape of uncondensed methyl borate. The tube leading into flask *C* shall be flared slightly to avoid plugging by separated salts. The apparatus shall be assembled with glassware and tubing resistant to heat and chemicals.[6]

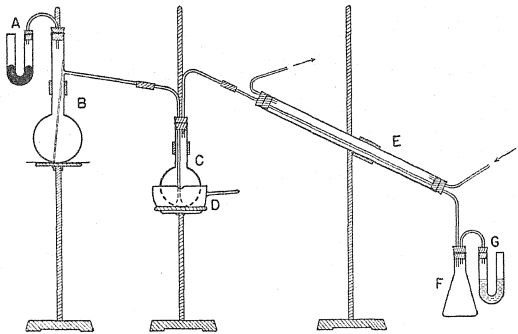


FIG. 2.—Apparatus for Distilling Boron.

Reagents

19. (*a*) *Methyl Alcohol.*—Alcohol free of boric acid and volatile organic acids shall be used. The more nearly anhydrous the alcohol, the better.

(*b*) *Mannite.*—Reagent grade, neutral.

(*c*) *Phenolphthalein Indicator.*—Dissolve 1 g. of phenolphthalein in 60 ml. of ethyl alcohol and dilute to 100 ml. with water.

(*d*) *Methyl Red Indicator.*—Dissolve 0.1 g. of methyl red in 60 ml. of ethyl alcohol and dilute to 100 ml. with water.

(*e*) *Calcium Chloride.*—Reagent grade, granular (8-mesh), anhydrous.

(*f*) *Boric Acid (0.1 N).*—Fuse pure boric acid in a platinum dish. While still warm, crush the melt and quickly place the fragments in a weighing tube. Dissolve 3.482 g. in 500 ml. of hot, recently boiled distilled water, cool, and dilute to exactly 1000 ml. Each milliliter of the solution contains 0.003482 g. of B_2O_3.

(*g*) *Sodium Hydroxide (approximately 0.1 N).*—Prepare the solution, as free of carbonates as possible, by diluting 6.5 ml. of a clear 50 per cent solution of NaOH with 1 liter of recently boiled distilled water. Standardize by titrating against the standard solution of boric acid exactly as described in Section 20.

(*h*) *Hydrochloric Acid (approximately 0.1 N).*—Prepare an approximately 0.1 *N* solution free of CO_2.

Procedure

20. (*a*) Mix 0.5000 g. of the prepared sample (Section 4) with exactly two times its weight of Na_2CO_3, and heat in a

[6] Pyrex glass or Kimble Glass No. N51a is satisfactory for this purpose. See *Industrial and Engineering Chemistry* (Anal. Ed.), Vol. 13, p. 419 (1941).

platinum crucible until a clear melt is obtained. Without removing it from the crucible, decompose the melt with diluted HCl (1:1) in calculated amount and added in small portions so as to avoid excessive frothing. While this is being done, the crucible should rest in a casserole, and the lid should be kept in place as much as possible. Toward the end, it may be necessary to heat a little, but care should be taken not to boil, since boric acid would be lost with the steam. Pour the solution into the flask *C*, Fig. 2, and rinse the crucible with a very small quantity of water.

(*b*) Add pure anhydrous $CaCl_2$, using about 1 g. for each milliliter of solution and dropping it through a paper funnel to keep the neck of the flask clean. Swirl the contents of the flask to allow the $CaCl_2$ to take up the water. Connect the flask to the apparatus, as shown in Fig. 2, and raise the casserole until the flask dips into the water but does not touch the bottom. Begin the distillation of alcohol from *B*, taking care that the "boiling tube" is functioning properly. After about 25 ml. of alcohol have condensed in *C*, heat the casserole with a small flame, keeping it hot enough to prevent condensation in the flask. When a distillate of 150 ml. has collected in flask *F*, replace the flask with an empty one, and continue the distillation.

(*c*) Add 1 drop of the phenolphthalein solution and 2 or 3 pellets of solid NaOH (about 0.25 g.) to the first distillate, stopper, and swirl until the NaOH has dissolved. The solution should be definitely alkaline; if not, add more NaOH. Transfer the contents of the flask to a platinum dish and heat gently on a steam bath to remove alcohol. Meanwhile a second distillate of 150 ml. will have collected. Add the contents of the protecting U-tube, make alkaline as with the first distillate, and transfer the solution to the platinum dish. Evaporate to dryness and dissolve the residue in about 10 ml. of hot water.

(*d*) Transfer the solution to a 200-ml. Erlenmeyer flask, and rinse with a minimum of hot water. The volume now should not exceed 25 ml. Add 5 drops of the methyl red solution (paranitrophenol may be substituted if desired) and then diluted HCl (1:1) drop by drop until the solution is acid, taking care not to add more than 1 drop in excess. Heat quickly to boiling and continue the boiling for 15 sec. Cool, add 5 drops of the phenolphthalein solution, and then drop by drop, add 0.1 *N* NaOH, until the red tint of the methyl red is discharged. One drop of 0.1 *N* HCl should restore the color, and 1 drop of 0.1 *N* NaOH should discharge it. Now add 1 g. of mannite, note the reading of the burette, and titrate until the phenolphthalein end point appears. Again add 1 g. of mannite and, if the color disappears, continue the titration until the color returns. Continue this process until the addition of 1 g. of mannite does not discharge the phenolphthalein end point.

(*e*) *Calculation.*—Correct the volume of 0.1 *N* NaOH required in passing from the methyl red end point to the phenolphthalein end point by subtracting the volume required in a blank run. Calculate the percentage of B_2O_3 as follows:

$$B_2O_3, \text{ per cent} = VA \times 200$$

where:

V = corrected volume in milliliters of NaOH solution required for titration from the methyl red end point to the phenolphthalein end point, and

A = B_2O_3 equivalent of the NaOH solution.

NOTE.—Results are likely to be low by 0.05 to 0.10 per cent in glasses containing 1 to 2 per

cent of B_2O_3 unless the boron in the residue in the distilling flask is recovered. To make this recovery, the residue in the flask must be filtered off, washed moderately with water, ignited, fused, carried through the procedure, and the result corrected for the blank on the reagents.

SULFUR TRIOXIDE

Reagent

21. *Barium Chloride Solution (10 per cent).*—Dissolve 100 g. of $BaCl_2 \cdot 2H_2O$ in 1000 ml. of water, allow to stand for 24 hr., and filter if the solution is not clear.

Procedure

22. (*a*) Transfer 5.00 g. of the prepared sample (Section 4) to a platinum dish, and add 25 ml. of water and 50 ml. of HF. Digest for 15 min. on the steam bath, and add 1 ml. of HNO_3, 15 ml. of $HClO_4$ (60 per cent), and 0.2 g. of $Mg(NO_3)_2 \cdot 6H_2O$. Evaporate on the steam bath until the HNO_3 and HF have been volatilized, and then heat on a hot plate at a low temperature so that light fumes of $HClO_4$ are evolved for about 5 min. Cool, wash the inside surface of the dish with a jet of water, and add 10 ml. of $HClO_4$ (60 per cent). Heat on a hot plate at a low temperature until nearly all of the $HClO_4$ has been evaporated.

(*b*) Cool, add 25 ml. of diluted HCl (5:95), and digest on the steam bath for 15 min. Add 150 ml. of hot water and 10 ml. of $BaCl_2$ solution. Digest on the steam bath for 1 hr. or more, and allow to stand at room temperature for 2 hr. or overnight. Filter through a tight paper, and wash with hot diluted HCl (1:99).

(*c*) Ignite the paper and precipitate slowly in a platinum crucible until all carbon is eliminated. Cool, and fuse the residue with 3 g. of Na_2CO_3. Dissolve the cooled melt in a small excess of diluted HCl (1:3), and digest the acid solution on the steam bath for 15 min. Then neutralize the excess acid with NH_4OH, using litmus as the indicator. Add 1 ml. of HCl, dilute the solution to 100 ml., heat to boiling, and add 5 ml. of $BaCl_2$ solution. Digest on the steam bath for 1 hr., and allow to stand at room temperature for 2 hr. or overnight. Filter, and wash as before. Ignite the paper and precipitate slowly at first until the carbon is eliminated, and finally at 1000 C. for 30 min. Cool and weigh.

(*d*) *Calculation.*—Calculate the percentage of SO_3 as follows:

$$SO_3, \text{ per cent} = \text{wt. of } BaSO_4 \times 6.86$$

PROCEDURES FOR ROUTINE ANALYSIS

These procedures for routine analysis admit of considerable variation, especially as regards the preparation of a master solution and the use of aliquot portions for several of the constituents. The procedures are designed for use under average laboratory conditions where it may be assumed that (*a*) several samples are to be analyzed simultaneously, with only a moderate supply of platinum ware, (*b*) all determinations are to be completed within one working day, and (*c*) the accuracy need not quite equal that obtainable with the referee methods.

Under certain conditions it may be of advantage to modify one or more of the steps in the procedure. For example:

(*1*) If the supply of platinum ware is not a factor, and if certain of the determinations are desired in a minimum of time, separate samples (0.1 g. and 0.5 g., respectively) may be decomposed for Na_2O and K_2O. In that case, a third sample (1.0 g.) is taken for BaO, R_2O_3, etc., and here decomposition may be effected by either HF and $HClO_4$ or HF and H_2SO_4, as desired. Under these conditions it becomes unnecessary to prepare a master solution, and it is possible to complete several of the determinations in an unusually short period of time.

(2) With glasses containing very small quantities of elements such as barium or aluminum, greater accuracy may be obtained by the use of a larger sample (2 g. or more), and the filtrate may be diluted to a definite volume at any desired point in the analysis and a suitable aliquot portion taken for the remaining determinations.

SILICON DIOXIDE

Procedure

23. (*a*) Intimately mix 0.5000 g. of the prepared sample (Section 4) with 1 g. of Na_2CO_3 in a 75-ml. platinum evaporating dish. Heat gradually at first over a flame, or in a muffle, and finally at a temperature high enough to give a *clear* melt. Allow the melt to solidify in a thin layer covering the bottom of the dish. Add about 10 ml. of diluted HNO_3 (1:1), and place the dish (covered) on a steam bath until all of the carbonates are decomposed. If desired, the cooled melt may be digested in water before the acid is added. Remove the dish, rinse the cover glass with a small quantity of water, and allow the solution to evaporate to dryness. Add 10 to 15 ml. of $HClO_4$ (60 per cent), and transfer the dish to a hot plate at about 200 to 220 C. Heat until fumes are evolved freely, then cover with a watch glass, and allow fuming to continue for 20 min. Sufficient $HClO_4$ should be present to prevent the contents of the dish from becoming dry. Remove the dish, cool, and dilute the contents with about 50 ml. of hot water. Stir to dissolve soluble salts, place the dish on a steam bath, and digest for 10 min. Pour the supernatant solution through a loose filter paper and flood the residue with about 10 ml. of hot diluted HCl (2:98). Stir and grind the solid lumpy material with a glass rod; then pour the solution through the filter. Transfer the precipitate completely to the filter by means of hot diluted HCl (2:98), using a rubber policeman, and continue washing the paper and precipitate with eight to ten small portions of the diluted HCl.

If desired the first dehydration can be made as described in the Procedures for Referee Analysis (Section 6 (*a*)); in this case add 10 to 15 ml. of $HClO_4$ (60 per cent) before the second evaporation. Evaporate the filtrate and washings in a 600-ml. beaker of heat-resistant glass[6] on a hot plate to a small volume; then heat at a reduced rate until copious fumes of $HClO_4$ appear. Rinse the inside of the beaker by means of 10 ml. of $HClO_4$ added from a pipette. Cover the beaker and continue heating for about 10 min. while dense fumes of $HClO_4$ are evolved.

(*b*) Allow the beaker to cool, and rinse the inside surface with about 50 ml. of distilled water. Digest for 10 min. on a water bath; then filter through a tight paper. Transfer the small precipitate completely to the paper, and wash paper and precipitate with ten portions of hot diluted HCl (2:98). Place both papers and precipitates in an untared 20-ml. platinum crucible, add 3 or 4 drops of diluted H_2SO_4 (1:1), and ignite slowly in a muffle furnace until the carbon is burned off and the residue is white. Cover and heat over a blast flame or in a muffle furnace at about 1200 C. for 20 min., cool in a desiccator, and weigh. Repeat ignition to constant weight. Moisten the residue with water, add about 10 ml. of HF and 1 drop of H_2SO_4, and evaporate to dryness on a hot plate. Cover the crucible and heat at approximately 1200 C. for 1 to 2 min. Cool in a desiccator and weigh. The loss in weight represents SiO_2.

(*c*) *Calculation.*—Calculate the percentage of SiO_2 as follows:

$$SiO_2, \text{ per cent} = \text{wt. of } SiO_2 \times 200$$

NOTE.—The net result obtained by this method is ordinarily slightly high (0.1 to 0.2 per cent) if B_2O_3 up to 2 per cent is present.

DETERMINATION OF BaO, R_2O_3, CaO, MgO, Na_2O, AND K_2O

Reagents

24. (*a*) *Standard Potassium Permanganate Solution.*—Dissolve 3.44 g. of $KMnO_4$ in distilled water, and dilute to 20 liters. This solution should stand for 3 to 4 months, and 1- to 3-liter portions of the clear supernatant liquid may then be siphoned off as required. Standardize against a solution containing a known amount of $CaCl_2$ by the procedure used in determining calcium (Section 28) rather than against sodium oxalate.

(*b*) *Standard Potassium Bromate (0.2 N) for MgO.*—Dissolve 120 g. of $KBrO_3$ in distilled water and dilute to 20 liters. Standardize against a known $MgCl_2$ solution by precipitating the quinolate as in the procedure for magnesium (Section 29), filtering, washing, dissolving with HCl, and titrating, using 0.1 *N* sodium thiosulfate for the back-titration.

(*c*) *Sodium Thiosulfate (0.1 N).*—Dissolve 25 g. of pure $Na_2S_2O_3 \cdot 5H_2O$ in recently distilled water and dilute to 1 liter. To obtain the equivalent of 1 ml. of thiosulfate in terms of standard bromate, add 10 ml. of bromate from a burette to 100 ml. of cool water containing 5 ml. of HCl; then add 10 ml. of KI (20 per cent), and titrate the liberated iodine with thiosulfate, using starch as indicator near the end point. The volume of bromate divided by the volume of thiosulfate (expressed to two decimal places) represents the volume of bromate equivalent to 1 ml. of thiosulfate.

(*d*) *Standard Magnesium Chloride Solution.*—Dissolve 1 g. of pure MgO in HCl and dilute to 2 liters. The exact concentration of MgO may be checked by a gravimetric determination (either as pyrophosphate or as quinolate ignited to MgO) on a 50-ml. aliquot portion.

(*e*) *8-Hydroxyquinoline Solution (5 per cent).*—To 5 g. of 8-hydroxyquinoline add 12 ml. of glacial acetic acid. Stir until dissolved, and dilute with 75 ml. of distilled water. Heat to 60 C. and stir until solution is complete, then filter into an amber bottle and dilute to 100 ml.

(*f*) *Potassium Iodide Solution.*—Dissolve 5 g. of KI in 25 ml. of distilled water and filter (if necessary). Store in an amber bottle. The solution deteriorates on long standing, and should be water-white when used.

(*g*) *Starch Indicator.*—Mix 2 g. of soluble starch with a little cold water and add to 100 ml. of boiling water. Cool and add 10 ml. of KI (25 per cent) and 10 ml. of NaOH (10 per cent). This solution keeps well over a fairly long period of time.

(*h*) *Zinc Uranyl Acetate Reagent for Na_2O.*—Transfer 100 g. of uranyl acetate, $UO_2(CH_3COO)_2 \cdot 2H_2O$, and 300 g. of zinc acetate, $Zn(CH_3COO)_2 \cdot 2H_2O$, to a bottle. Add 90 ml. of acetic acid (30 per cent) and then 810 ml. of warm water. Shake at intervals for a few hours. If the solution remains clear, add a few crystals of NaCl so as to saturate the solution with respect to sodium zinc uranyl acetate. Filter small portions of the reagent as required.

(*i*) *Chloroplatinic Acid Solution for K_2O.*—Dissolve 15 g. of chloroplatinic acid, $H_2PtCl_6 \cdot 6H_2O$, in 50 ml. of water containing 5 ml. of diluted HCl (1:1). One milliliter will contain about 0.1 g. of platinum, equivalent to 0.05 g. of K_2O.

(*j*) *Barium Chloride Solution (10 per cent).*—Dissolve 100 g. of $BaCl_2 \cdot 2H_2O$ in 1000 ml. of water, allow to stand for 24 hr., and filter if the solution is not clear.

Preparation of Solution

25. Transfer 2 g. of the prepared sample (Section 4) to a 75-ml. platinum dish, moisten with water, and add 1 ml.

of HNO_3, 10 ml. of $HClO_4$ (60 per cent), and about 25 ml. of HF (48 per cent). Evaporate on a steam bath to small volume, and then add about 10 ml. more of HF. Again evaporate to small volume. Transfer the dish to a hot plate (200 to 215 C.) and allow to fume until about one half of the $HClO_4$ is expelled. Examine for particles of undecomposed glass, and, if any are noted, add 5 ml. more of HF (48 per cent), and again evaporate to copious fumes. Cool the dish, and rinse down the inside surface with 5 ml. of $HClO_4$ (60 per cent) added from a pipette. Return the dish to a hot plate, and allow to fume briefly. Cool the dish, add about 0.5 g. of boric acid, and rinse down the inside of the dish with a little distilled water. Again evaporate to fumes, and continue the heating until most of the acid is eliminated, but not to the point where the residue appears dry. (A deep yellow to orange color at this point indicates that the glass contains chromium.) Add 50 ml. of hot water and 2 ml. of HCl and stir until the soluble salts are entirely dissolved. (The solution may be turbid if the glass contains both BaO and SO_3.) (Note). Rinse into a 200-ml. volumetric flask, cool to room temperature, and dilute to the mark. Mix thoroughly, and quickly pipette out the following aliquot portions:

(*1*) 100 ml. (1 g. of sample) into a 600-ml. beaker for BaO, "R_2O_3," CaO, MgO.

(*2*) 10 ml. (0.1 g. of sample) into a 50-ml. beaker for Na_2O.

(*3*) 50 ml. (0.5 g. of sample) into the platinum dish for K_2O.

NOTE.—Large amounts of BaO and SO_3 occurring together may cause difficulty through separation of $BaSO_4$. In such case, decompose a separate 1 g. sample of glass for each determination of BaO, R_2O_3, CaO, and MgO.

Determination of BaO

26. (*a*) Neutralize the reserved 100-ml. aliquot portion (Section 25) with NH_4OH, using methyl red as indicator. Then add 1 ml. of HCl. Dilute to about 150 ml., cover the beaker, heat to boiling, and add 10 ml. of diluted H_2SO_4 (1:9) from a pipette. Continue the boiling for 10 min.; then transfer to the steam bath and digest for 45 min. Filter through a tight paper, transfer the precipitate completely, and wash on the paper with four portions of warm water. Reserve the filtrate and washings for the determination of R_2O_3. Ignite the precipitate slowly in a tared porcelain crucible in a muffle furnace until the carbon is burned; then heat at about 1000 C. for 15 min. Cool in a desiccator, and weigh. (If the residue is not white, add a drop of H_2SO_4, heat gently until the acid is vaporized, then again ignite, cool, and weigh.)

(*b*) *Calculation.*—Calculate the percentage of BaO as follows:

$$\text{BaO, per cent} = \text{wt. of } BaSO_4 \times 65.7$$

Determination of R_2O_3

27. (*a*) Add 5 ml. of HCl to the filtrate from the BaO determination (Section 26) and boil for 5 min. to make sure that chromium is reduced to the trivalent state. Add diluted NH_4OH (1:1) drop by drop until the color of methyl red just changes to yellow, cover the beaker, and allow the contents to boil for 2 min. Filter immediately through a loose paper, rinse the beaker twice, and wash the precipitate on the paper four times with small quantities of hot, neutral ammonium chloride solution (2 per cent). Reserve the filtrate for the determination of CaO.

(*b*) Ignite the paper and precipitate in a tared platinum crucible, first in a muffle furnace until the carbon is eliminated, and finally for 10 min. at about 1200 C. Cool in a desiccator, and weigh.

(*c*) *Calculation.*—Calculate the percentage of R_2O_3 (including Al_2O_3, Cr_2O_3,

Fe_2O_3, TiO_2, ZrO_2, P_2O_5, and V_2O_5) as follows:

$$R_2O_3, \text{ per cent} = \text{wt. of precipitate} \times 100$$

Determination of CaO

28. (*a*) *Volumetric Method.*—Heat the reserved filtrate from the R_2O_3 separation (Section 27) to boiling in a covered beaker and add slowly 20 ml. of saturated ammonium oxalate solution from a pipette. Continue boiling for 5 min., transfer to a steam bath, and digest for 20 min. Filter through a tight paper, rinse the beaker two or three times with warm water, and wash the paper and precipitate four times with warm water (60 C.). Reserve the filtrate for the determination of MgO. Unfold the paper, and rinse the oxalate precipitate back into the original beaker by means of a stream of warm water. Discard the paper. Dilute to about 100 ml., add 20 ml. of diluted H_2SO_4 (1:1), heat nearly to boiling, and titrate with standard $KMnO_4$ solution to the first permanent pink. Calculate the percentage of CaO as follows:

$$\text{CaO, per cent} = VA \times 100$$

where:

V = volume in milliliters of $KMnO_4$ solution required for titration, and

A = CaO equivalent of $KMnO_4$ solution.

(*b*) *Gravimetric Method.*—Alternatively, the calcium oxalate may be ignited to the oxide and weighed as described in Section 11.

Determination of MgO

29. (*a*) Dilute to 500 ml. the filtrate reserved from the CaO separation (Section 28), add 10 ml. of 8-hydroxyquinoline solution (5 per cent) (1 ml. = 0.006 g. of MgO), and warm to 60 C. Add diluted NH_4OH (1:1) drop by drop, while stirring vigorously, until a precipitate starts to form, then add, drop by drop, an excess of 10 ml. of NH_4OH. Allow to settle for at least 20 min. Filter on a fritted glass crucible of medium porosity with suction. Transfer the precipitate by means of a rubber policeman, and wash four times with cold diluted NH_4OH (1:200). Discard the filtrate.

(*b*) *Volumetric Method.*—Dissolve the precipitate (Paragraph (*a*)) by means of 30 ml. of warm (60 C.) diluted HCl (1:1) added in small quantities and drawn through the filter with suction; wash thoroughly with hot water. Collect the solution in a 500-ml. Erlenmeyer flask, and dilute to about 100 ml. Cool the flask to about 20 C. Add 1 g. of KBr crystals, and then add standard $KBrO_3$ solution from a burette until a slight excess is present, as shown by the formation of a blue color when a drop of the solution is brought in contact with starch iodide paper. The excess of bromate should not exceed 2 to 3 ml. Allow to stand for about 1 min. to complete the bromination. Add 5 ml. of fresh KI solution (20 per cent) (resulting in a brown color or brown precipitate) and titrate with standard thiosulfate until most of the brown color has disappeared. Add 1 ml. of starch solution, and continue the titration to the disappearance of the blue color. Calculate the bromate equivalent of the thiosulfate, and subtract from the original volume of bromate to find the net volume equivalent to the MgO. Calculate the percentage of MgO as follows:

$$\text{MgO, per cent} = VA \times 100$$

where:

V = net volume in milliliters of bromate solution required for titration of the MgO, and

A = MgO equivalent of the bromate solution.

(*c*) *Gravimetric Methods.*—The following gravimetric methods may be used for the routine determination of MgO:

(*1*) The magnesium precipitate (Para-

graph (*a*)) may be dried to constant weight at 130 to 140 C. on the fritted glass filter and weighed as magnesium hydroxyquinolate ($Mg(C_9H_6NO)_2$), if desired.

MgO, per cent = wt. of $Mg(C_9H_6NO)_2 \times 12.9$

(*2*) The magnesium precipitate (Paragraph (*a*)) may be caught on a filter paper and converted to MgO by igniting gradually until the carbon is burned and finally heating at 1000 C.

MgO, per cent = wt. of MgO × 100

Determination of Na_2O

30. (*a*) Evaporate the reserved 10-ml. aliquot portion (Section 25) in a 50-ml. beaker to about 1 ml. on a hot plate. Cool, and add slowly 20 ml. of the zinc uranyl acetate reagent while stirring. Continue the stirring for 3 min. Allow the precipitate to settle for at least 20 min. at room temperature, then filter on a weighed fritted glass filter of medium porosity with suction. Extreme temperatures should be avoided; temperatures controlled at approximately 21 C. (70 F.) are preferable. Transfer the precipitate completely by means of several small quantities of the precipitating solution contained in a wash bottle, using a rubber policeman. Wash the precipitate on the filter twice with 2-ml. quantities of the same solution, then six times with small volumes of acidified (1 ml. of acetic acid in 100 ml. of alcohol) 95 per cent ethyl alcohol that has been saturated at room temperature with sodium zinc uranyl acetate. Allow the wash solution to drain through completely between washes. Finally, wash the precipitate twice with 5-ml. portions of *anhydrous* ether, added from a pipette. Continue to draw air through the filter for a minute or two after the precipitate appears dry, wipe off the crucible, set it aside near the balance for 10 to 15 min., and weigh. The empty crucible may be washed and dried in the same manner at the start and weighed as soon as it reaches room temperature. As a check on the presence of significant amounts of foreign insoluble matter in the precipitate, the crucible can be put under suction, the sodium zinc uranyl acetate dissolved in hot water, and the crucible reweighed after final washing with ether.

(*b*) *Calculation.*—Calculate the percentage of Na_2O as follows:

Na_2O, per cent = wt. of precipitate × 20.15

Determination of K_2O

31. (*a*) Evaporate the reserved 50-ml. aliquot portion (Section 25) to dryness in the 75-ml. platinum dish, and heat on the hot plate to eliminate free $HClO_4$. Perchlorates need not be destroyed, but all free $HClO_4$ should be eliminated. Cool, rinse down the inside of the dish with a little water, and add about 1 ml. of HCl and 0.5 ml. of chloroplatinic acid solution. Evaporate nearly to dryness on the steam bath so that the residue solidifies on cooling. Add about 20 ml. of ethyl alcohol (80 per cent), stir to dissolve all soluble salts, crush the residue with the flattened end of a glass rod, and filter through a fritted glass filter of medium porosity with suction. Wash the residue once or twice by decantation, transfer to the filter, and wash two or three times more on the filter with ethyl alcohol (80 per cent). Using a 100-ml. beaker as the receiver, rinse out the dish with hot water, pour it through the filter, and wash thoroughly to dissolve all potassium chloroplatinate. Add 2 ml. of HCl and a strip of magnesium ribbon to the solution, and warm while stirring until all of the platinum is reduced. The reduction with magnesium is conveniently made by wrapping the ribbon around the end of the stirring rod. Add more magnesium if necessary, and finally boil the solution for a few minutes. After settling, the solution will be color-

less if the reduction of the chloroplatinate is complete. Filter and wash the platinum several times with hot water. Ignite in a porcelain crucible in a muffle, gradually at first, and finally at 1000 C. for 5 min. Cool in a desiccator, and weigh.

(*b*) *Calculation.*—Calculate the percentage of K_2O as follows:

$$K_2O, \text{ per cent} = \text{wt. of platinum} \times 96.5$$

BORIC OXIDE

Reagents

32. (*a*) *Bromcresol Purple Indicator (0.04 per cent).*—Treat 0.10 g. of bromcresol purple with 18.5 ml. of 0.01 *N* NaOH, and dilute to 250 ml. with water. (Methyl red or *p*-nitrophenol may be used instead of bromcresol purple.)

(*b*) *Phenolphthalein Indicator (0.5 per cent).*—Dissolve 1.0 g. of the reagent in 100 ml. of 95 per cent ethyl alcohol, and dilute to 200 ml. with water.

(*c*) *Hydrochloric Acid (approximately 0.1 N).*—Prepare an approximately 0.1 *N* HCl solution free of CO_2.

(*d*) *Sodium Hydroxide (approximately 1 N).*—Prepare an approximately 1 *N* NaOH solution free of CO_2 and B_2O_3. Solutions of NaOH attack borosilicate glass. Consequently all such solutions should be freshly prepared, or stored in bottles containing no boron.

(*e*) *Sodium Hydroxide (0.05 N).*—Prepare an approximately 0.05 *N* NaOH solution free of CO_2, and standardize against the National Bureau of Standards standard sample 84*a* of acid potassium phthalate.

Procedure

33. (*a*) Intimately mix 1.000 g. of the prepared sample (Section 4) and 2.0 g. of Na_2CO_3 in a platinum crucible, and heat until a clear melt is obtained. Rotate the crucible while cooling so that the melt solidifies in a thin layer. Add 10 ml. of diluted HCl (1:1), cover, and allow to digest at room temperature with occasional stirring with a glass rod until the melt is disintegrated.

(*b*) Transfer to a 100-ml. glass-stoppered cylinder, rinse the crucible several times with small quantities of water, and dilute to 25 ml. Add 25 ml. of absolute ethyl alcohol and 50 ml. of anhydrous ether. Stopper the graduate, and mix the contents by vigorous intermittent shaking over a 5 min. period. Finally, allow the cylinder to stand for an hour or more, or until the two liquid layers have separated completely. Note and record the volumes of the two layers. Pipette out 50 ml. of the upper (ether-alcohol) layer, and transfer to a 200-ml. Erlenmeyer flask.

(*c*) Add 1 drop of bromcresol purple indicator, then add 1 *N* NaOH drop by drop, while shaking vigorously, until a permanent blue end point is reached. Add 1 ml. of 0.5 per cent phenolphthalein solution, and continue the addition of the NaOH to a pink end point. Finally, add an excess amounting to about three times the volume used between the two end points (never less than 1 ml.). Add about 50 ml. of water, and warm the solution carefully on the steam bath until the ether layer has evaporated; then transfer the flask to a hot plate and allow to boil until most of the alcohol has been eliminated. Cool, add diluted HCl (1:1) drop by drop until the solution is just acid, then add 1 *N* NaOH to faint alkalinity.[7] Treat with 0.1 *N* HCl until definitely acid (5 to 10 drops excess), warm on the steam bath, attach to a vacuum line, and allow to boil under reduced pressure for a few minutes to eliminate dissolved gases.

(*d*) Cool to room temperature, add 0.05 *N* NaOH from a burette until the

[7] With glasses containing zinc, this procedure results in its complete precipitation as the hydroxide, which may be eliminated by filtration. This step also removes all of the iron, which is likely to be troublesome if present in abnormal amounts.

color changes from yellow to blue, and record the burette reading. Add about 1 g. of mannite, and continue the titration to a permanent pink color that is not destroyed by further additions of mannite.

(*e*) *Calculation.*—The volume of NaOH solution required between the blue and the pink end points (Paragraph (*d*)) provides a measure of the boric acid contained in the aliquot portion of the ether layer. It is then possible to calculate the amount of boric acid contained in the entire ether layer, and from a knowledge of the distribution ratio, the quantity in the aqueous layer. The boric oxide content of the sample is calculated as follows:

$$B_2O_3, \text{ per cent} = V \times \left(A + \frac{B}{K}\right) \times 0.00348$$

where:

V = volume in milliliters of 0.05 N NaOH required for titration of 50 ml. of the alcohol-ether layer,

A = volume in milliliters of the alcohol-ether layer,

B = volume in milliliters of the aqueous layer, and

K = partition coefficient.

NOTE.—The value of K (partition coefficient) is taken as 0.36 for ordinary laboratory temperatures of about 25 C. If the extraction is made at a room temperature of 30 C. or higher, the value becomes 0.34.

(*f*) *Blank Titration.*—The percentage of B_2O_3 shall be corrected by a blank on the reagents. This may be done conveniently by repeating the entire procedure (Paragraphs (*a*) to (*e*)) with a glass (such as the National Bureau of Standards standard sample No. 80) known to contain no boron.

FLUORINE

Reagents

34. (*a*) *Thorium Nitrate (0.1 N).*—Dissolve 13.8 g. of $Th(NO_3)_4 \cdot 4H_2O$ in water and dilute to 1 liter. Standardize against the standard fluorine solution described in Paragraph (*b*) by titrating as described in Section 35.

(*b*) *Standard Fluorine Solution.*—Dissolve 0.442 g. of pure NaF in 1 liter of water. One milliliter of this solution will contain 0.0002 g. of fluorine. If pure NaF is not available, it may be prepared by treating pure $NaHCO_3$ with an excess of pure HF in a tubulated Gooch crucible or in a small platinum dish, evaporating the excess of HF, and heating to about 600 C. in an atmosphere of nitrogen. The NaF should be cooled in nitrogen and stored over a good desiccant.

(*c*) *Buffer Solution.*—Dissolve 9.45 g. of monochloracetic acid and 2.00 g. of NaOH in 100 ml. of water.

(*d*) *Sodium Alizarin Sulfonate Indicator.*—Prepare an aqueous 0.05 per cent solution.

Procedure

35. (*a*) Fuse 1 g. of the prepared sample (Section 4) with 4 g. of Na_2CO_3 in a platinum crucible, and allow the melt to solidify in a thin layer. Nearly fill the crucible with water, and allow to digest on the steam bath until the melt is thoroughly disintegrated. Transfer the disintegrated melt to a 250-ml. Claissen flask, rinsing the crucible several times with water. The flask should contain eight to ten glass beads to prevent bumping. Add cautiously 35 ml. of H_2SO_4 while the flask is kept cool in running water. Close the flask with a two-hole rubber stopper through which passes a thermometer and a 4-mm. glass tube extending nearly to the bottom of the flask. Connect the tube to a 2-liter Florence flask filled with water, which serves as a steam generator. Close the connection between the generator and distilling flask by means of a pinchcock.

(*b*) Connect the distilling flask to the

condenser, and distill until the temperature of the liquid reaches 135 C. In the meantime, heat the water in the Florence flask to boiling, and when a temperature of 135 C. is reached, admit steam into the Claissen flask. Adjust the rate of flow of the steam (by regulating the burners under the two flasks) so as to keep the volume and temperature of the contents of the Claissen flask about constant. Continue the distillation until about 400 ml. of distillate has collected. Add phenolphthalein and sufficient 1 *N* NaOH to the distillate to make it distinctly alkaline, and evaporate to about 100 ml.

(*c*) Cool the solution, then add 10 drops of sodium alizarin sulfonate indicator and diluted HCl (1:1). until slightly acid. Neutralize with 1 *N* NaOH, then make faintly acid with 0.1 *N* HCl. Add 1 ml. of the buffer solution and titrate with 0.1 *N* thorium nitrate to a faint, permanent pink. A blank determination should be made, using a glass that contains no fluorine (such as the National Bureau of Standards standard sample No. 128).

(*d*) *Calculation.*—Calculate the percentage of fluorine as follows:

$$\text{Fluorine, per cent} = VF \times 100$$

where:

V = volume in milliliters of thorium nitrate solution required for titration, and

F = fluorine equivalent of the thorium nitrate solution.

SULFUR TRIOXIDE

Procedure

36. (*a*) Decompose 3 g. of the prepared sample (Section 4) by treatment with $HClO_4$ and HF exactly as described in Section 25 and transfer the solution to a 600-ml. beaker. Dilute to about 200 ml., neutralize with NH_4OH, then add HCl until an excess of 2 ml. is present. Cover the beaker, heat to boiling, and add 10 ml. of $BaCl_2$ (10 per cent) drop by drop from a pipette. Continue boiling for 10 min., transfer to a steam bath, and digest for 45 min. Filter through a tight paper, transfer the precipitate completely, and wash paper and precipitate with four portions of warm water. Ignite slowly in a tared porcelain crucible until the carbon is burned, and finally heat at about 1000 C. for 10 min. Cool in a desiccator, and weigh.

(*b*) *Calculation.*—Calculate the percentage of SO_3 as follows:

$$SO_3\text{, per cent} = \text{wt. of } BaSO_4 \times 11.43$$

Standard Method of

HYDROSTATIC PRESSURE TEST ON GLASS CONTAINERS[1]

A.S.T.M. Designation: C 147 - 43

ADOPTED, 1943.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 147; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method covers the test procedure for determining the breaking strength of narrow-mouth glass containers when subjected to a uniform internal hydrostatic pressure. The method is intended to determine the uniformity of glass bottles and the serviceability of pressure containers.

Apparatus

2. The apparatus,[3] a suitable type of which is illustrated in Fig. 1, shall embody the following principles:

(*a*) The bottles to be tested shall be held by a split collar so designed that the bottle is not clamped but is suspended from the "bead of the finish."

(*b*) A seal shall be provided which will be watertight at the pressure to be applied.

(*c*) An automatically controlled timing mechanism shall be built into the machine so that the bottle will be under pressure for 1 ± 0.02 min.

(*d*) The apparatus shall be designed so that the weight is applied directly by gravity without any intermediate cams or levers.

Sampling

3. The method of sampling a group of bottles of a given type shall be in accordance with Paragraphs (*a*) to (*c*) for the various situations described.

(*a*) *Sampling in Manufacturing Plant During Continuous Production:*

(*1*) For continuous routine checking of quality to insure that a predetermined standard of internal pressure strength is being maintained in the glassware, a sample of at least one bottle from each cavity or mold shall be taken every 3 hr. from the exit end of the annealling lehr. If the margin between the strength of

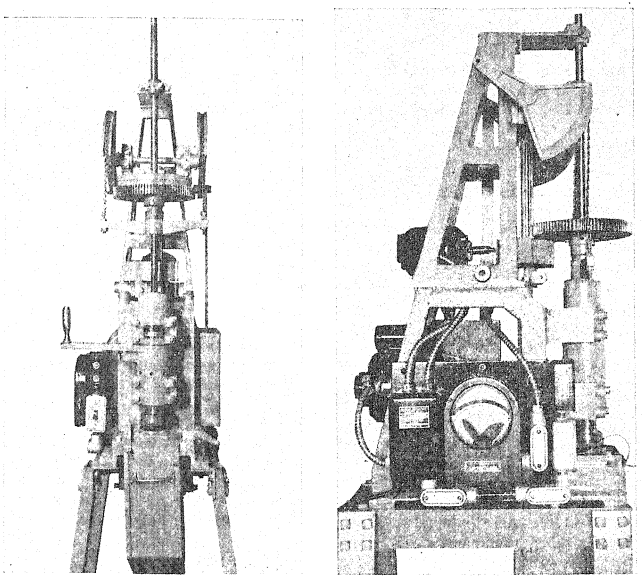
[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-14 on Glass and Glass Products.

[2] Prior to adoption as standard, this method was published as tentative from 1939 to 1943, being revised in 1940 and 1943.

[3] Six-head and single-head automatic sustained pressure testing machines developed by the Preston Laboratories meet these requirements. Detailed working drawings of the apparatus are available at a nominal cost from the American Society for Testing Materials, 260 S. Broad St., Philadelphia 2, Pa.

the glassware and the predetermined standard becomes low because of radical changes in the operations and adjustments of the machine, samples shall be taken at intervals sufficiently short to insure that all necessary information does not know which cartons will be opened by the sampler.

(*3*) For a detailed test of new mold designs or of radical changes in machine operations a sample of 10 bottles from each mold or cavity shall be required



Front View Side View

FIG. 1.—Pressure Testing Machine for Hydrostatic Test with Automatic Lift.

concerning variations in strength is be- obtained.

(*2*) For continuous routine checking of quality, including handling and packing, one bottle from each cavity or mold shall be taken every 3 hr. from cartons packed by the inspector or the packer and in such a manner that the packer for test. In case the total number of molds shall be more than 10, a total of 100 bottles distributed among the molds shall be required for the test. The bottles shall be taken from the exit end of the annealing lehr.

(*b*) *Sampling New or Unused Aged Bottles in Original Containers in Ware-*

house, Freight Car, or Truck.—A sample of at least 100 bottles shall be taken in accordance with the principles of random sampling. If the group of bottles is contained in more than 100 cartons or crates, not more than one bottle shall be taken from each container, and random sampling shall be applied to the various containers in selecting the ones to be opened. If the bottles are contained in less than 100 cartons or crates, insofar as possible, equal numbers of bottles shall be taken from each container. If the bottles are in bulk rather than in cartons or crates, the principles of random sampling shall be applied to the various parts of the several rows, layers, or tiers.

(*c*) *Sampling Stock or Group of Used or Filled Bottles in a Bottling, Packaging, or Distribution Plant, such Bottles Having Been Shuffled with Respect to Original Containers and Having Complicated and Unknown Histories.*—Whether in cartons or in racks, layers, rows, or lines, a sample of at least 100 bottles shall be taken in accordance with the principles of random sampling, and in accordance with Paragraph (*b*). If the group or stock contains bottles manufactured by more than one firm and of various vintages, the sample which is taken shall be large enough to represent proportionately all manufacturers and all vintages. Whenever possible, random samples of 100 bottles from each manufacturer and of each vintage shall be tested. Bottles bearing no manufacturer's trade-mark or other identification shall be tested separately.

Procedure

4. (*a*) The bottles shall be nearly filled with water, the permissible air space being governed by the length of plunger of the machine. The bottles shall then be placed in the split collar and the specified test pressure applied. If the bottle does not burst, this pressure will be automatically released at the end of 1 min. The test shall be continued as described in Paragraphs (*b*) to (*d*), depending on the nature of the sample.

(*b*) *Pass Test.*—The "pass test" at a specified pressure as described in Paragraph (*a*) is sufficient for the routine testing of samples from continuous production in a manufacturer's plant (see Section 3 (*a*) (*1*)).

(*c*) *Progressive Test (50 per cent Level).*—All samples obtained in accordance with Section 3 (*a*) (*2*), (*b*), and (*c*) shall be given a "progressive test" in which the test described in Paragraph (*a*) is repeated, the pressure being increased 25 psi. each time until the 50 per cent level is reached; that is, the pressure required to burst at least 50 per cent of the bottles.

(*d*) *Progressive Test (Total).*—As an alternative to the progressive 50 per cent level test described in Paragraph (*c*), the progressive test may be continued until all of the bottles break.

Report

5. The report shall include the following information, depending on the particular procedure used in the test. In all cases the detailed technique and criterion followed in taking the random sample shall be reported.

(*a*) *Pass Test in Accordance with Section 4 (b)*:

(*1*) Time of taking sample,

(*2*) Number of samples from each mold, if more than one,

(*3*) The test procedure, and

(*4*) Whether or not each bottle passed the test.

(*b*) *Progressive Test in Accordance with Section 4 (c)*:

(*1*) The initial breakage level or pressure and the number of bottles which broke at that pressure, and

(*2*) The pressure required to break 50 per cent of the bottles, interpolated from the data to the nearest 5 psi. Report this figure as the "50 per cent breakage level" or simply the "50 per cent level."

(*c*) *Progressive Test in Accordance with Section 4 (d):*

(*1*) The bursting pressures, and

(*2*) The average bursting pressure (subtract 12.5 psi. to correct for the 25 psi. loading interval).

Standard Method of

POLARISCOPIC EXAMINATION OF GLASS CONTAINERS[1]

A.S.T.M. Designation: C 148 - 43

ADOPTED, 1943.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 148; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method for the polariscopic examination of glassware describes the procedure for determining the relative annealing stress in glass containers by comparison with reference standards. The method is intended to determine whether the annealing of the glass is commercially satisfactory.

NOTE.—The method is not intended to evaluate the cordiness or cord stresses due to composition inhomogeneities. The ring section examination[3], which is frequently used as a special means for investigating glass quality, is not yet included in this method.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Polariscope.*—A polariscope conforming to the following requirements: High brightness of field—at least 90 foot lamberts at the point where the glassware is held for examination and at least 50 foot lamberts at the edge of the field as observed with the polarizing element and protective element in operating position. The degree of polarization of the field at all points shall be not less than 99.0 per cent. The field shall be of sufficient size to permit the examination of glass containers 25 cm. (10 in.) in height at one viewing. A sensitive tint plate standardized to retard approximately a full wave length of light of 565 mμ shall be used.

(*b*) *Reference Standards.*—A set of standard disks of known internal stress.

NOTE.—Recently the Glass Container Association has prepared such a set of standard disks.[4] These disks or others prepared with at least the same degree of precision should be used for the comparison. These reference standards consist of a set of five disks, individually checked for path difference, so mounted that they can be combined additively and their total effect observed in comparison with that of a container. The optical path difference in each case is 22.8 mμ at points ¼ in. from edge. The methods of standardizing and value of errors have been completely described by Mr. T. D. Green in the private communications of the Glass Container Association.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-14 on Glass and Glass Products.

[2] Prior to adoption as standard, this method was published as tentative from 1939 to 1943, being revised in 1940 and 1943.

[3] V. C. Swicker, "The Ring-Section Examination of Glass Containers," *Bulletin* Am. Ceramic Soc., Vol. 18, No. 4, pp. 143 to 147 (1939).

[4] The standard disks are obtainable from the Hartford-Empire Co., Hartford, Conn.

Sampling

3. The method of sampling shall be in accordance with Paragraphs (*a*) to (*c*) for the various situations described.

(*a*) *Sampling in Manufacturing Plant During Continuous Production.*—For continuous routine checking of quality to insure that a predetermined standard of temper is being maintained in the glassware, a sample of glassware shall be taken across the lehr at least once every 3 hr., such sample being large enough to represent any possible differences in the annealing treatment across the lehr and large enough to represent the various types of bottles simultaneously in the lehr. Usually 6 bottles will suffice. If the margin between the temper and the predetermining standard becomes low because of radical changes in operation, samples shall be taken at intervals sufficiently short to insure that all necessary information concerning variations in temper is being obtained.

(*b*) *Sampling New or Unused Aged Bottles in Original Containers in Warehouse, Freight Car, or Truck.*—A sample of at least 100 bottles shall be taken in accordance with the principles of random sampling. If the group of bottles is contained in more than 100 cartons or crates, not more than one bottle shall be taken from each container, and random sampling shall be applied to the various containers in selecting the ones to be opened. If the bottles are contained in less than 100 cartons or crates, insofar as possible, equal numbers of bottles shall be taken from each container. If the bottles are in bulk rather than in cartons, or crates, the principles of random sampling shall be applied to the various parts of the several rows, layers, or tiers.

(*c*) *Sampling Stock or Group of Used or Filled Bottles in a Bottling, Packaging, or Distribution Plant, Such Bottles Having Been Shuffled with Respect to Original Cartons and Having Complicated and Unknown Histories.*—Whether in cartons or in racks, layers, rows, or lines, a sample of at least 100 bottles shall be taken in accordance with the principles of random sampling, and in accordance with Paragraph (*b*). If the group or stock contains bottles manufactured by more than one firm and of various vintages, the sample which is taken shall be large enough to represent proportionately all manufacturers and all vintages. Whenever possible, random samples of 100 bottles from each manufacturer and of each vintage shall be tested. Bottles bearing no manufacturer's trade-mark or other identification shall be tested separately.

Procedure

4. (*a*) *Examination of Base of Cylindrical Bottles.*—In determining the grade of annealing or the temper of the base of an ordinary cylindrical flint bottle, the bottle shall be viewed through the base. The maximum broad color pattern (Note) in the base of the bottle shall be compared with the maximum color in various numbers of the standard disks by using the disks in numeric sequence ("stacked" one on top of the other) to determine whether the maximum amount of color in the bottle base is less than that in one disk, less than that in two and greater than one, greater than two, etc. It is seldom possible to obtain an exact match of the color of the bottle with the reference standards. The temper of the bottle shall then be recorded in accordance with the following rules:

Rule.—When the color of a bottle is greater than that of N disks but less than $N + 1$ disks, the temper is $N + 1$; that is, corresponding numerically to the larger number of disks, as follows:

Temper 1.....less than 1 disk
Temper 2.....less than 2, greater than 1 disks
Temper 3.....less than 3, greater than 2 disks
Temper 4.....less than 4, greater than 3 disks
Temper 5.....less than 5, greater than 4 disks
Temper 6.....greater than 5 disks

NOTE.—The color observed by the procedure described in Paragraphs (*a*), (*b*), and (*c*) should be the broad color patterns produced by annealing stresses, and this temper is considered the "lehr temper." Local stresses clearly due to cords or composition inhomogeneities should be distinguished from the broad pattern and ignored in the assignment of the temper grading.

(*b*) *Examination of Square, Oval, and Irregular Shapes.*—In determining the temper of square or oval and other irregular shapes, the polariscopic examination shall be made at the curve or corner that shows the most color and the temper shall be recorded in accordance with the rule given in Paragraph (*a*).

(*c*) *Examination of Sidewalls.*—In determining the temper of the sidewalls of any bottle, the maximum color of any part of the sidewall shall be matched with the maximum color in any part of the disks, and the temper shall be recorded in accordance with the rule given in Paragraph (*a*).

Report

5. The report shall include a record of the temper gradings, and information concerning observed areas of localized strain arising from a cordy condition of the glass.

Standard Method of

THERMAL SHOCK TEST ON GLASS CONTAINERS[1]

A.S.T.M. Designation: C 149 - 43

ADOPTED, 1943.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 149; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test for determining the relative resistance of commercial glass containers (bottles and jars) to thermal shock is intended to apply to all types of glass containers which are required to withstand temperature changes (thermal shock) in service such as in washing, pasteurization, or "hot pack" processes, or in being transferred from a warm to a colder medium or *vice versa*.

NOTE.—It is recommended that glassware subjected to this thermal shock test be not later subjected to the hydrostatic pressure test, because lower pressure strengths will be obtained.

Apparatus

2. (*a*) The apparatus[3] consists essentially of a basket for holding the glassware and two tanks, one containing hot water and one containing cold water. An automatic mechanism for immersing and transferring the basket of bottles from the hot to the cold bath shall be provided and shall be so geared that exactly 15 sec. will be consumed in the transfer. A suitable type of apparatus is illustrated in Fig. 1.

(*b*) A device shall be provided to maintain the temperature of the baths within plus or minus 1 F. of the specified temperatures. Indicating controllers which operate solenoid valves to admit hot water to the hot tank or cold water to the cold tank are recommended. Otherwise, a dial thermometer should be attached and the valves operated manually.

Sampling

3. The method of sampling a group of bottles of a given type shall be in accordance with Paragraphs (*a*) to (*c*) for the various situations described.

(*a*) *Sampling in Manufacturing Plant During Continuous Production:*

(*1*) For continuous routine checking of quality to insure that a predetermined standard of internal pressure strength

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-14 on Glass and Glass Products.

[2] Prior to adoption as standard, this method was published as tentative from 1939 to 1943, being revised in 1940 and 1943.

[3] Detailed drawings of satisfactory apparatus for this test are available at a nominal cost from the American Society for Testing Materials, 260 S. Broad St., Philadelphia 2, Pa.

is being maintained in the glassware, a sample of at least one bottle from each cavity or mold shall be taken every 3 hr. from the exit end of the annealing lehr. If the margin between the strength of the glassware and the predetermined standard becomes low because of radical changes in the operations and adjustments of the machines, samples shall be taken at intervals sufficiently short to insure that all necessary information concerning variations in strength is being obtained.

(2) For continuous routine checking of quality, including handling and packing, one bottle from each cavity or mold shall be taken every 3 hr. from cartons packed by the inspector or the packer and in such a manner that the packer does not know which cartons will be opened by the sampler.

(3) For a detailed test of new mold designs or of radical changes in machine operations a sample of 10 bottles from each mold or cavity shall be required for test. In case the total number of molds shall be more than 10, a total of 100 bottles distributed among the molds shall be required for the test. The bottles shall be taken from the exit end of the annealing lehr.

(*b*) *Sampling New or Unused Aged Bottles in Original Containers in Warehouse, Freight Car, or Truck.*—A sample of at least 100 bottles shall be taken in accordance with the principles of random sampling. If the group of bottles is contained in more than 100 cartons or crates, not more than one bottle shall be taken from each container, and random sampling shall be applied to the various containers in selecting the ones to be opened. If the bottles are contained in less than 100 cartons or crates, insofar as possible, equal numbers of bottles shall be taken from each container. If the bottles are in bulk rather than in cartons, or crates, the principles of random sampling shall be applied to the various parts of the several rows, layers, or tiers.

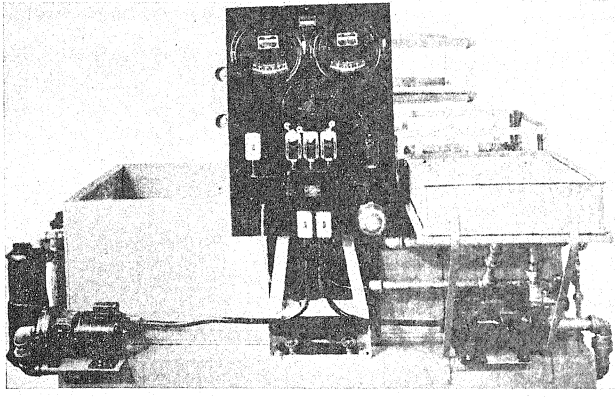


FIG. 1.—Automatic Thermal Shock Testing Machine.

(*c*) *Sampling Stock or Group of Used or Filled Bottles in a Bottling, Packaging, or Distribution Plant, Such Bottles Having Been Shuffled with Respect to Original Containers and Having Complicated and Unknown Histories.*—Whether in cartons or in racks, layers, rows, or lines, a sample of at least 100 bottles shall be taken in accordance with the principles of random sampling, and in accordance with Paragraph (*b*). If the group or stock contains bottles manufactured by more than one firm and of various vintages, the sample which is taken shall be large enough to represent proportionately all manufacturers and all vintages. Whenever possible, random samples of 100 bottles from each manufacturer and of each vintage shall be tested. Bottles bearing no manufacturer's trade-mark or other identification shall be tested separately.

Procedure

4. (*a*) The temperature of the baths shall be adjusted so that the cold bath is at 70 F. and the hot bath is at a temperature hotter than the cold bath by the specified increment (in most cases this increment will be 75 F. for the first immersion). When the temperatures of the baths are within plus or minus 1 F. of those specified, the baskets shall be filled with bottles and immersed in the hot bath, allowed to soak for 5 min., then transferred to the cold bath and immersed for 30 sec., and then removed from the bath. The 5-min. immersion in the hot bath shall be controlled within 3 sec. and the time required for transfer from the hot to the cold bath shall be 15 ± 0.33 sec. During the test the apparatus shall be protected from drafts in a sheltered laboratory. The test shall then be continued according to one of the alternatives in Paragraph (*b*) and (*c*), depending on the nature of the sample.

(*b*) *Pass Test.*—The "pass test" at a specified increment or temperature differential as described in Paragraph (*a*) is sufficient for the routine testing of samples from continuous production in a manufacturer's plant (see Section 3 (*a*) (*1*)).

(*c*) *Progressive Test (50 per cent Level).*—All samples obtained in accordance with Section 3 (*a*) (*2*), (*b*), and (*c*) shall be given a "progressive test" in which the test described in Paragraph (*a*) is repeated, the temperature differential being increased 5 F. each time by increasing the temperature of the hot water, until at least 50 per cent of the bottles are broken.

NOTE.—Bottles which have not broken in the test should not be used for further testing.

Report

5. The report shall include the following information, depending on the particular procedure used in the test. In all cases the detailed technique and criterion followed in taking the random sample shall be reported.

(*a*) *Pass Test in Accordance with Section 4 (b):*

(*1*) Time of taking sample,

(*2*) Number of samples taken from each mold, if more than one,

(*3*) The temperature differential employed in the test, and

(*4*) Whether or not each bottle passed the test.

(*b*) *Progressive Test in Accordance with Section 4 (c):*

(*1*) The initial breakage level and the number broken, and

(*2*) The thermal shock differential required to break 50 per cent of the bottles, interpolated to the nearest 1 F. This figure is the "50 per cent breakage level" or simply the "50 per cent level."

Standard Method of

FLEXURE TESTING OF GLASS[1]

DETERMINATION OF MODULUS OF RUPTURE

A.S.T.M. Designation: C 158 - 43

ADOPTED, 1943.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 158; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test covers the procedure for determining the modulus of rupture of glass, that is, the stress in flexure which a specimen of the glass develops at rupture. In practice the modulus of rupture is usually taken as the stress at which the specimen breaks in the flexure test.

Apparatus

2. (*a*) *Testing Machine.*—The loading device shall be sufficiently adjustable to give a uniform rate of increase of stress in the specimen of 10,000 ± 1,000 psi. per min. (7.0 ± 0.7 kg. per sq. mm. per min.). The apparatus shall be suitably insulated from any external vibration.

(*b*) *Bearing Edges.*—For the support of the test specimen a set of two knife-edge bearing surfaces approximately $\frac{1}{16}$ in. in radius and made of brass or mild steel shall be provided. The supports shall be spaced 8 in. between centers. The load shall be applied through a third knife-edge bearing located centrally between the supports and mounted in such a manner that the applied load acts always in a vertical line. For the testing of flat specimens, the central bearing and one of the outer bearings shall be pivoted laterally to compensate for minor irregularities of the test specimen.

Test Specimens of Rods

3. (*a*) *Preparation of Specimens.*—Information concerning the method of manufacture of the glass and the rods shall be supplied with the samples. Any special treatment given the material shall be indicated. At least 30 specimens shall be used for one test (Note). Test specimens shall be at least 10 in. in length and $\frac{3}{8} \pm \frac{1}{32}$ in. in diameter. Although most rods are of elliptical section, the maximum and minimum diameters of the specimen shall not differ by more than 0.020 in., nor shall either diameter vary by more than 0.005 in. in 10 in. The specimens shall be straight within $\frac{1}{8}$ in. in 10 in., and the curve, if present, shall be in one plane only.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-14 on Glass and Glass Products.

[2] Prior to adoption as standard, this method was published as tentative from 1940 to 1943, being revised in 1943.

(*b*) *Examination of Specimens.*—After careful inspection, test specimens containing any obvious defect shall be discarded. At least ten of the specimens shall be examined for strain. If any of these is not within the requirements prescribed in the Appendix, the remainder of the specimens shall be examined and those exceeding the strain test limit shall be rejected.

NOTE.—In view of the large variation encountered in measurements of the modulus of rupture, not only a large number but a wide range of samples is essential. For example, in the case of rods the specimens should be cut from a number of different lengths of cane; specimens of flat glass should be taken from several sheets, or at least from several regions of a single sheet. Otherwise, the results may be representative of only a small sample.

Test Specimens of Flat Glass

4. (*a*) *Preparation of Specimens.*—Test specimens of flat glass shall be cut from sheet stock with a diamond or a steel wheel. Both longitudinal cuts shall be on the same original surface, and none of the original edge of the sheet shall be used as the longitudinal side of a specimen. The end cuts may be made on either surface. The direction of cutting approximately half the total number of specimens shall be perpendicular to the direction of cutting the remainder. At least 30 specimens shall be used for one test (Note, Section 3). Specimens shall be approximately 10 in. in length and $1\frac{1}{2} \pm \frac{1}{32}$ in. in width. Specimens of plate glass shall be $\frac{1}{4} \pm 0.030$ in. in thickness. Drawn glass shall be designated in commercial or calipered thickness within plus or minus 0.010 in. The thickness of any specimen shall not vary by more than plus or minus 0.005 in.

(*b*) *Examination of Specimens.*—Any specimen may be rejected for defects considered likely to cause undue weakness. Specimens showing surface scratches, poor cutting or splinters, reentrant angles or irregularities on the side opposite the cutter mark shall be rejected. Annealing shall be such that the stress at the center, viewed edgewise, does not exceed 200 psi. (Appendix).

Procedure

5. Specimens having cut edges shall be broken with the cutter marks on the side under compression. The permissible initial load shall not exceed that corresponding to a stress of approximately 4000 psi. The specimen shall be bent until fractured under a constant loading rate such that the rate of increase of stress is 10,000 ± 1,000 psi. per min.

NOTE.—No specimen may be rejected during test because of low flexural strength.

Calculation and Report

6. (*a*) *Calculation.*—The initial stress, rate of increase of stress, and modulus of rupture shall be calculated as follows:

Stress due to initial load:

$$R_0 = \frac{w_0}{w} \times R$$

Rate of increase of stress:

$$S = \frac{R - R_0}{t}$$

Modulus for elliptical rod specimens:

$$R = 20.36 \frac{w}{bd^2}$$

Modulus for rectangular specimens:

$$R = 12 \frac{w}{bd^2}$$

where:

R = modulus of rupture in pounds per square inch,

R_0 = stress due to initial load in pounds per square inch,

S = rate of increase of stress in pounds per square inch,
w_o = initial load in pounds,
w = breaking load in pounds,
b = width of specimen in inches,
d = thickness of specimen in inches, and
t = time from start of continuous loading rate to rupture in minutes.

NOTE.—Metric units may be used by substitution of the proper constants.

(*b*) *Report.*—The report shall include the data itemized in Paragraph (*a*) and the maximum, minimum, and average value of the modulus of rupture for each group of similar specimens. The standard deviation of the modulus of rupture shall also be obtained in accordance with the A.S.T.M. Manual on Presentation of Data.[3]

APPENDIX

METHOD FOR MEASURING STRAIN

A1. Strains shall be measured with a calibrated wedge or Babinet compensator.

A2. The stress optical coefficient shall be determined for unknown glass, but may be taken as 1 mμ per inch of light path which is equivalent to 2.18 psi. (1 mμ per centimeter of light path, equivalent to 0.389 kg. per sq. cm.) for common soda lime window, plate, and bottle glass.

A3. Laths of glass shall not have more than 200 psi. tension at the middle nor more than 400 psi. compression at the surface. This is equivalent respectively to 90 mμ and 180 mμ per inch of light path in common glass.

A4. Specimens of rods may be examined by viewing through a diameter distant at least four diameters from the nearest end. The *apparent* tension at the center shall not exceed 133 psi. (equivalent to 61 mμ per inch of diameter for common soda lime glass). As an alternative, specimens of rods may be examined by cutting a piece from the end and measuring the stress at the circumference by viewing it axially. The stress shall not exceed 400 psi. (equivalent to 180 mμ per inch of length for common soda lime glass).

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 453 (1933). Also available as a separate publication.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: A6-1925

Standard Specifications for

DRAIN TILE[1]

A.S.T.M. Designation: C 4 – 24

ADOPTED, 1914; REVISED, 1916, 1921, 1924.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 4; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover three classes of drain tile as follows:

Farm Drain Tile, intended for ordinary private drainage work on farms, for moderate sizes and depths,

Standard Drain Tile, intended for ordinary district land drainage at moderate depths,

Extra-Quality Drain Tile, intended for district land drainage, for considerable depths and where an extra quality is desired.

Basis of Purchase

2. The purchaser shall specify the class or classes of tile to be supplied, whether Farm Drain Tile, Standard Drain Tile, or Extra-Quality Drain Tile. Standard Drain Tile shall be supplied where no other advance selection is stated.

Basis of Acceptance

3. (*a*) The acceptability of drain tile shall be determined (1) by the results of the chemical and physical tests hereinafter specified, and (2) by visual inspection, to determine whether the tile complies with the specifications as to dimensions, shape, and freedom from visible external and internal defects.

(*b*) The acceptance of drain tile as satisfactorily meeting one of these two general requirements shall not be construed as in any way waiving the other.

MATERIALS AND MANUFACTURE

Materials

4. (*a*) These specifications shall apply to drain tile made of shale, fire clays, or surface clays and to drain tile made of concrete.

(*b*) By shale is meant a thinly stratified, consolidated, sedimentary clay[3] with well-marked cleavage parallel to the bedding.

(*c*) By fire clay is meant a sedimentary clay[3] of low flux content.

NOTE.–It is usually associated with coal measures.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-6 on Drain Tile.

[2] Editorially revised in 1939.

[3] These descriptions of the terms "surface clay," "fire clay," and "shale" are based upon the following definition for the term "clay":

Clay.—An earthy or stony mineral aggregate consisting essentially of hydrous silicates of alumina, plastic when sufficiently pulverized and wetted, rigid when dry, and vitreous when fired at a sufficiently high temperature.

(*d*) By surface clay is meant an unconsolidated, unstratified clay,[3] occurring on the surface.

(*e*) By concrete is meant a suitable mixture of portland cement, mineral aggregates, and water, hardened by hydraulic chemical reaction.

(*f*) If the purchaser desires to exclude any of these materials he shall so specify in advance. All materials used shall be first-class of their kind and suitable for the purpose.

Manufacture

5. The method of manufacture shall be such as to insure excellence of product and uniformity in quality.

CHEMICAL TESTS AND REQUIREMENTS

Chemical Tests and Requirements

6. (*a*) The purchaser may specify special chemical requirements as to resistance of the tile to chemical action in cases where soils or drainage waters have marked acid or alkaline character, or are of abnormally high temperature, and may prescribe chemical tests of the tile to ascertain whether these special requirements are met. Without a special agreement in advance, no drain tile shall be rejected by reason of its composition as determined by ultimate chemical analysis.

(*b*) The presence in drain tile of visible grains or masses of caustic lime, iron pyrites, or any other minerals which are known to cause slaking or disintegration of the tile, shall be construed as a valid ground for rejection, unless satisfactory proof be submitted that the tile is permanent and durable, and that the objectionable minerals are not present in quantity or condition to work damage.

PHYSICAL TESTS

Physical Tests

7. The physical tests of drain tile shall include strength tests and absorption tests, and may also include freezing and thawing tests when specified by the purchaser in advance or when called for by the manufacturer or other seller as provided in Sections 34, 35, 47, and 52.

Selection of Specimens

8. The specimens of tile shall all be selected at the factory or at the shipping destination, or at the trench, at the option of the purchaser. The selection shall be made by a competent inspector employed by the purchaser. The inspector shall divide the tile into subclasses if lack of uniformity in any important particular warrants such division, and shall select enough representative specimens of tile from each sub-class for a complete set of standard physical tests.

Number and Cost of Specimens

9. A standard physical test shall comprise tests of five individual tiles. Specimens of tile may be selected by the inspector in such number as he judges necessary to determine fairly the quality of all the tile. The manufacturer or other seller shall furnish specimens of tile without separate charge up to 0.5 per cent of the whole number of tile, and the purchaser shall pay for all in excess of that percentage at the same rate as for other tile.

Strength Tests

Test Specimens

10. The specimens of tile for the strength test shall be unbroken, full-size tile.

Moisture Condition of Specimens

11. The walls of the tile shall be, at the time of testing, as thoroughly wet as will result from completely covering with hay, cloth, or similar absorbent material, and keeping the covering wet for not less than 12 hr.

Temperature Condition of Specimens

12. No specimen of tile shall be exposed to water or air temperatures lower than 40 F. from the beginning of wetting until tested. Frozen tile shall be completely thawed before the wetting begins.

Weighing

13. Each specimen shall be weighed on reliable scales just prior to testing, and the weights shall be reported.

Application of Load

14. Any machine or hand method which will apply the load continuously, or in increments not exceeding 5 per cent of the estimated total breaking load, may be used in making the test. The tile shall not be allowed to stand under load longer than is required for observing and recording the loads. All solid parts of the bearing frames and bearing blocks shall be so rigid that the distribution of the load will not be affected appreciably by the deformation of any part. All bearings and the specimens of tile shall be so accurately centered as to secure a symmetrical distribution of the loading on each side of the center of the tile in every direction.

Choice of Method

15. The purchaser shall choose (1) sand bearing method, (2) two-edge-bearing method, or (3) three-edge-bearing method, for use in making strength tests of drain tile (see Sections 18, 19, and 20).

Calculations and Report

16. The test results shall be calculated and reported, in pounds per linear foot of tile, in terms of the "ordinary supporting strength."[4]

The ordinary supporting strength shall be calculated by multiplying the test breaking loads by the following factors: For sand bearings, 1.00; for two-edge bearings, 1.50; for three-edge bearings, 1.50.

The results of the strength tests shall be reported separately for each of the five individual specimens of tile constituting a standard test, together with the average.

Modulus of Rupture

17. The modulus of rupture may or may not be calculated and reported, at the option of the purchaser. When reported it shall be calculated as follows:[5]

$$M = 0.20\, r \frac{W}{12} \quad \ldots\ldots\ldots (1)$$

$$f = \frac{6M}{t^2} \quad \ldots\ldots\ldots\ldots (2)$$

where:

M = maximum bending moment in wall in pound-inches per inch of length,
r = radius of middle line of tile wall in inches,
W = ordinary supporting strength, calculated as prescribed in Section 16, in pounds per linear foot of tile,
f = modulus of rupture in pounds per square inch, and
t = thickness of tile wall in inches.

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[4] The "ordinary supporting strength," when calculated as specified in Section 16, is approximately equal to the actual supporting strength of a tile when laid in a ditch by the "ordinary" method. See Note under Table II.

[5] The coefficient of 0.20 in Eq. 1 approximates the value found by theoretical analysis and also that determined by extended tests.

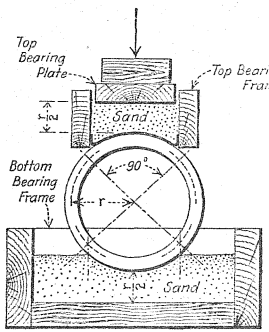


FIG. 1.—Sand Bearings.

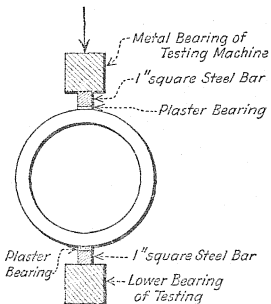
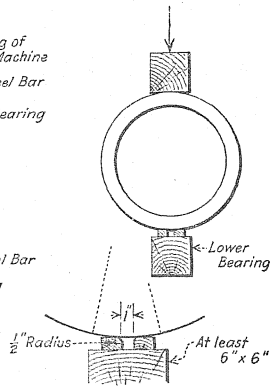


FIG. 2.—Two-Edge Bearings.



Detail of Lower Bearing.

FIG. 3.—Three-Edge Bearings.

Five-eighths of the weight of the tile per linear foot for sand bearings, or three-fourths for two-edge or three-edge bearings, shall be added to W in calculating the maximum bending moment M, when such addition exceeds 5 per cent of W. The value of t used shall be the average thickness of the wall at the top of the tile or that at the bottom, selecting the lesser of the two.

Sand Bearing Method

18. (*a*) When the sand-bearing method is used (see Fig. 1), the ends of each specimen of tile shall be accurately marked in quarters of the circumference prior to the test. Specimens shall be carefully bedded in sand, above and below, for one-fourth the circumference of the tile measured on the middle line of the wall. The depth of bedding above and below the tile at the thinnest points shall be one-half the radius of the middle line of the wall.

(*b*) The sand used shall be clean, and shall be such as will pass a 4760-micron (No. 4) sieve.

(*c*) The top bearing frame shall not be allowed to come in contact with the tile nor with the top bearing plate. The upper surface of the sand in the top bearing shall be struck level with a straightedge, and shall be covered with a rigid top-bearing plate, the lower surface of which is a true plane, made of heavy timbers or other rigid material, capable of distributing the test load uniformly without appreciable bending. The test load shall be applied at the exact center of this top bearing plate, in such a manner as to permit free motion of the plate in all directions. For this purpose a spherical bearing is preferred, but two rollers at right angles may be used. The test may be made without the use of a testing machine by piling weights directly on a platform resting on the top bearing plate, provided, however, that the weights shall be piled symmetrically about a vertical line through the center of the tile, and that the platform shall not be allowed to touch the top bearing frame.

(*d*) The frames of the top and bottom bearings shall be made of timbers so heavy as to avoid appreciable bending by the side pressure of the sand. The interior surfaces of the frames shall be dressed. No frame shall come in contact with the tile during the test. A strip of cloth may be attached, if desired, to the inside of the upper frame on each side along the lower edge, to prevent the escape of sand between the frame and the tile.

Two-Edge-Bearing Method

19. (*a*) When the two-edge-bearing method is used (see Fig. 2) the ends of each specimen of tile shall be accurately marked in halves of the circumference prior to the test.

(*b*) The tile to be tested shall be supported by a metallic knife bearing 1 in. wide and extending the length of the tile. Before the tile is placed, a fillet of plaster of Paris and sand 1 in. wide, and thick enough to compensate for all the inequalities of the tile barrel, shall be cast on the surface of the knife-edge bearing. The tile shall be placed upon the fillet while the plaster of Paris is still somewhat plastic. The load shall be applied through an upper knife bearing of the same size and length as the lower bearing. A plaster of Paris fillet 1 in. wide shall be cast along the length of the crown of the tile to equalize the lower bearing before the upper one is brought into contact.

(*c*) Both of the bearings shall be sufficiently rigid to transmit and receive uniform loads throughout their lengths without deflection, and shall be so attached to the machine as to transmit

and receive the maximum stresses produced by the test without lost motion, vibration, or sudden shock.

Three-Edge-Bearing Method

20. When the three-edge-bearing method is used (see Fig. 3), the ends of each specimen of tile shall be accurately marked in halves of the circumference prior to the test. The lower bearings shall consist of two wooden strips with vertical sides, having their interior top corners rounded to a radius of approximately $\frac{1}{2}$ in. The strips shall be straight, and shall be securely fastened to a rigid block with their interior vertical sides 1 in. apart. The upper bearing shall be a wooden block, straight and true from end to end. The test load shall be applied through the upper bearing block in such a way as to leave the bearing free to move in a vertical plane passing midway between the lower bearings. In testing a tile which is "out of straight," the lines of the bearings chosen shall be from those which appear to give most favorable conditions for fair bearings.

Absorption Test

Test Specimens

21. Not less than three separate test specimens from each of five separate tiles shall be taken as a "standard sample" for the absorption test. Of the three specimens from each tile, one shall be taken from one end, another from the opposite end, and the third shall be taken from the middle portion of the tile. Each specimen shall be of from 12 to 20 sq. in. in area, measured upon the exterior or convex side, and shall be as nearly square as the nature of the material will readily permit. The specimens shall be obtained by breaking the tile, and shall be apparently sound, solid pieces of the wall of the tile, and shall not show cracks or fissures or shattered edges due to the shock of breaking or cutting. The specimens may be obtained from the broken pieces of the tiles used in the strength test, if the restrictions as to the size and location of the specimens can be duly observed. The specimens shall be so marked as to permit the identity of each one to be ascertained at any stage of the test.

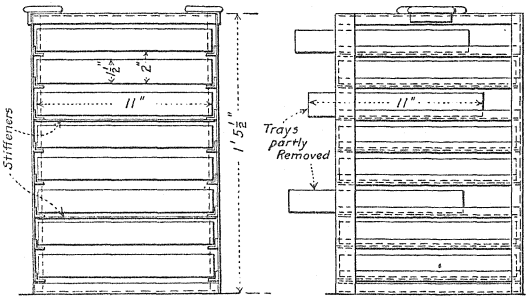
Drying Test Specimens

22. Preparatory to the absorption test, all specimens shall be first weighed and then dried in a drier or oven, at a temperature of not less than 110 C. (230 F.) for not less than 3 hr. After removal from the drier, the specimens shall be allowed to cool to a temperature of 20 to 25 C. (68 to 77 F.) and reweighed. If the specimens were apparently dry when taken, and the second weight closely checks the first, the specimens shall be considered dry. If the specimens were known to be wet when taken, they shall be placed in the drier for a further drying treatment of 2 hr, and reweighed. If the third weight checks the second, the specimens shall be considered dry. In case of any doubt, the specimens must be redried for 2-hr. periods until check weights are obtained.

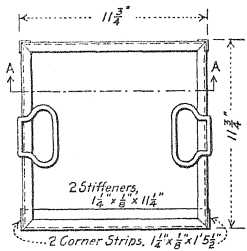
Weighing and Reweighing

23. The balance used shall be sensitive to 0.5 g. when loaded with 1 kg., and weighings shall be read at least to the nearest gram. Where other than metric weights are used, the same order of accuracy must be obtained.

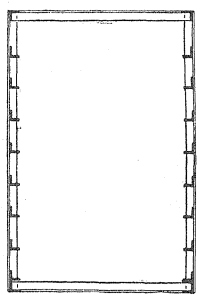
In reweighing after immersion, the specimens shall be removed from the water, allowed to drain for not more than 1 min., the superficial water removed by towel or blotting paper, and the specimens at once put upon the balance.



Front Elevation. Side Elevation.



Top View, Trays Removed.



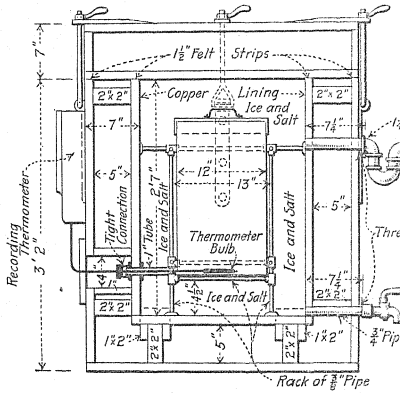
Section A-A, Trays Removed.

Notes:

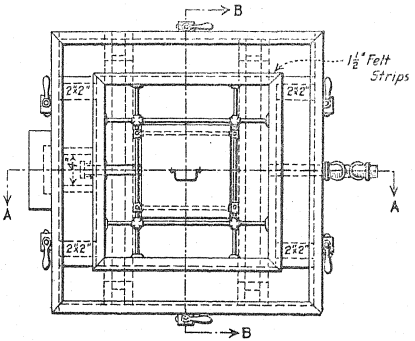
Rack Constructed of ⅝" x ⅝" x ⅛" Galvanized Angles, except as Noted. All Connections Riveted or Soldered.

Trays, 11" x 11" x 1½" outside, Made of No. 17 Galvanized Steel.

FIG. 4.—Suggested Plans for Freezing Crate and Trays.



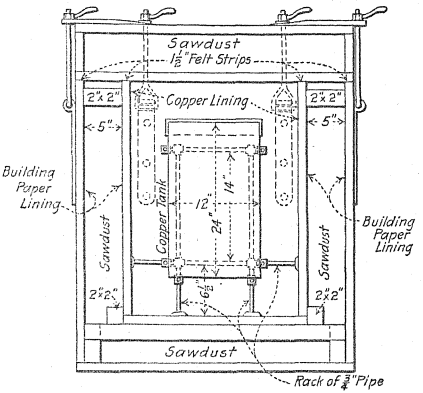
Section A-A.



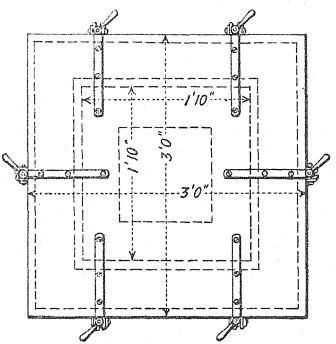
Plan View,
Top Removed.

NOTE.—Box to be constructed of seasoned white pine, free from

FIG. 5.—Suggested Plans for Freezing



Section B-B



Plan View, Top in Place

defects, or other suitable timber 1 by 6 in., unless otherwise specified.

Box, Using Salt and Ice for Freezing.

Immersion of Test Specimens

24. Specimens after weighing shall be placed in a suitable woven-wire receptacle, packed tightly enough to prevent jostling, covered with distilled water or rain water, raised to the boiling point and boiled for 5 hr., and then cooled in water to a final temperature of 10 to 15 C. (50 to 59 F.).

Calculations and Report

25. The test results shall be calculated as percentages of the initial dry weight, carried to the nearest first decimal place. The results shall be reported separately for each individual specimen, together with the mean of the fifteen or more specimens comprising the standard sample, the maximum and the minimum single observations entering into the mean, and the variation between the maximum and the minimum of the three specimens of each tile represented in the standard sample.

Freezing-and-Thawing Tests

Test Specimens

26. The test specimens employed in making the freezing-and-thawing test shall be another set selected as specified in Section 21 for the absorption test.

Drying Test Specimens

27. The specimens shall be dried as specified in Section 22.

Weighing and Reweighing

28. The same scales and weights as are specified in Section 23 for the absorption test or others of equivalent sensitivity and accuracy shall be employed for the weighings required in the freezing-and-thawing test. The same procedure in weighings and reweighing as specified in Section 23 shall be followed.

Immersion of Test Specimens

29. The specimens shall be immersed for 72 hr. in water having a temperature of 18 to 24 C. (65 to 75 F.) and then weighed as specified in Section 23.

Freezing and Thawing

30. When the specimens have been weighed after saturation with water, they shall be returned to the water and kept immersed until the freezing test is begun. For freezing, they shall be placed with their concave faces upward in watertight metal trays, suitably mounted in a rigid metal crate,[6] and immersed in ice water until the specimens have attained substantially the temperature of the water, after which the water shall be drawn down to a depth of $\frac{1}{2}$ in. in each tray. The crate shall then be lifted as a whole, without disturbing the specimens, and placed in the freezing apparatus.

Freezing shall be performed in a quiet atmosphere, free from perceptible natural or artificial currents. If artificial freezing apparatus is employed,[7] the apparatus shall have sufficient heat-absorbent capacity to enable the temperature of the freezing chamber to be brought to −10 C. (+14 F.) or below, within 30 min. after the introduction of the specimens. The temperature in the freezing apparatus shall not fall lower than − 20 C. (−4 F.). The freezing shall be continued until the water in the trays is frozen solid. Exposure to freezing conditions in excess of this requirement shall be considered as without significance.

At the conclusion of freezing under the specified conditions, the crate of

---

[6] Fig. 4 shows a crate and trays suitable for use in the box for artificial freezing illustrated in Fig. 5.

[7] Artificial freezing will generally be necessary. It may be conducted in a commercial zero (Fahrenheit) refrigerating room, or in an artificial freezing box similar to the one shown in Fig. 5 in which zero temperature (Fahrenheit scale) can readily be produced by the use of salt and ice.

specimens shall be withdrawn and at once immersed in water at a temperature of 18 to 24 C. (65 to 75 F.) in a special receptacle of proper size. Heating shall be continued so that the water will regain the required temperature as soon as practicable after the specimens are immersed. A temperature of 18 to 24 C. (65 to 75 F.) shall then be maintained for not less than 2 hr. At the conclusion of the thawing treatment, the crate of specimens shall be inspected and the condition of each sample after each thawing shall be noted in the records.

Method of Determining Failure

31. Failure under the freezing-and-thawing treatment shall be considered to be reached when:

(*a*) The specimens show superficial disintegration or spalling with loss of weight of more than 5 per cent of the initial dry weight; or,

(*b*) The specimens are badly cracked in other than lamination planes; or,

(*c*) The specimens show evident serious loss of structural strength.

TABLE I.—PHYSICAL TEST REQUIREMENTS FOR DIFFERENT CLASSES OF DRAIN TILE.

CAUTION.—Drain tile meeting these requirements are not necessarily safe against cracking in ditches. For safe requirements as against cracking, see Table II.

Internal Diameter of Tile, in.	Farm Drain Tile				Standard Drain Tile				Extra-Quality Drain Tile			
	Minimum Average Ordinary Supporting Strength, lb. per linear ft.	Maximum Average Absorption by Standard Boiling Test, per cent			Minimum Average Ordinary Supporting Strength, lb. per linear ft.	Maximum Average Absorption by Standard Boiling Test, per cent			Minimum Average Ordinary Supporting Strength, lb. per linear ft.	Maximum Average Absorption by Standard Boiling Test, per cent		
		Shale and Fire-Clay Tile	Surface-Clay Tile	Concrete Tile		Shale and Fire-Clay Tile	Surface-Clay Tile	Concrete Tile		Shale and Fire-Clay Tile	Surface-Clay Tile	Concrete Tile
4	800	11	14	12	1200	9	13	10	1600	7	11	9
5	800	11	14	12	1200	9	13	10	1600	7	11	9
6	800	11	14	12	1200	9	13	10	1600	7	11	9
8	800	11	14	12	1200	9	13	10	1600	7	11	9
10	800	11	14	12	1200	9	13	10	1600	7	11	9
12	800	11	14	12	1200	9	13	10	1600	7	11	9
15	1000	11	14	12	1300	9	13	10	1600	7	11	9
18					1400	9	13	10	1800	7	11	9
21					1550	9	13	10	2100	7	11	9
24					1700	9	13	10	2400	7	11	9
27					1850	9	13	10	2700	7	11	9
30					2000	9	13	10	3000	7	11	9
33					2150	9	13	10	3300	7	11	9
36					2300	9	13	10	3600	7	11	9
39					2450	9	13	10	3900	7	11	9
42[a]	(Not Permitted)	(Not Permitted)	(Not Permitted)	(Not Permitted)	2600	9	13	10	4200	7	11	9

[a] Larger sizes recommended as standard are as follows: 45, 48, 54, and 60 in.

NOTE.—When the freezing-and-thawing test is made as provided in Section 7, the number of freezings and thawings to be endured shall be as follows: For farm drain tile, 24; for standard drain tile, 36; for extra-quality drain tile, 48.

PHYSICAL TEST REQUIREMENTS

Physical Test Requirements

32. The different classes of drain tile shall conform to the physical test requirements prescribed in Table I.

Absorption of Tile Made of Mixed Clays

33. Drain tile made of mixtures of surface clays with other clays shall conform to the absorption requirements for surface-clay tile in Table I when the proportion of surface clay is 75 per cent or more and to the requirements for

TABLE II.—STANDARD ORDINARY SUPPORTING STRENGTHS OF DRAIN TILE FOR ORDINARY SAND AND FOR THOROUGHLY WET CLAY DITCH FILLING MATERIALS.

STRENGTHS IN POUNDS PER LINEAR FOOT

Height of Fill above Top of Tile, ft.	Breadth of Ditch at Top of Tile.																			
	1 ft.				2 ft.				3 ft.				4 ft.				5 ft.			
	Method of Laying Pipe.				Method of Laying Pipe.				Method of Laying Pipe.				Method of Laying Pipe.				Method of Laying Pipe.			
	Ordinary.		First Class.		Ordinary.		First Class.		Ordinary.		First Class.		Ordinary.		First Class.		Ordinary.		First Class.	
	Ditch Filling Material.		Ditch Filling Material.		Ditch Filling Material.		Ditch Filling Material.		Ditch Filling Material.		Ditch Filling Material.		Ditch Filling Material.		Ditch Filling Material.		Ditch Filling Material.		Ditch Filling Material.	
	Sand.	Clay.	Sand.	Clay.	Sand.	Clay.	Sand.	Clay.	Sand.	Clay.	Sand.	Clay.	Sand.	Clay.	Sand.	Clay.	Sand.	Clay.	Sand.	Clay.
2	265	280	220	235	615	635	510	530	970	990	810	830	1 330	1 350	1 110	1 130	1 690	1 710	1 410	1 430
4	400	450	335	375	1 050	1 120	880	935	1 750	1 820	1 460	1 520	2 450	2 540	2 040	2 110	3 160	3 250	2 640	2 710
6	470	545	390	455	1 370	1 500	1 140	1 250	2 370	2 530	1 980	2 110	3 410	3 580	2 840	2 980	4 460	4 640	3 720	3 870
8	505	605	420	505	1 600	1 790	1 330	1 490	2 870	3 110	2 390	2 590	4 220	4 490	3 510	3 740	5 596	5 890	4 660	4 910
10	525	640	440	535	1 760	2 010	1 470	1 680	3 270	3 610	2 730	3 010	4 900	5 290	4 080	4 410	6 590	7 020	5 490	5 850
12	535	660	445	550	1 880	2 190	1 570	1 820	3 600	4 030	3 000	3 355	5 480	6 000	4 570	5 009	7 460	8 030	6 220	6 690
14	540	675	450	560	1 960	2 320	1 640	1 930	3 850	4 380	3 210	3 650	5 980	6 620	4 980	5 520	8 230	8 950	6 850	7 460
16	545	680	455	565	2 030	2 420	1 690	2 020	4 060	4 670	3 390	3 890	6 400	7 160	5 330	5 970	8 890	9 770	7 410	8 150
18	545	685	455	570	2 070	2 500	1 720	2 090	4 220	4 920	3 520	4 100	6 750	7 630	5 630	6 360	9 480	10 520	7 900	8 770
20	545	690	455	575	2 100	2 560	1 750	2 140	4 370	5 130	3 640	4 280	7 050	8 000	5 880	6 720	9 990	11 200	8 330	9 330
22	545	690	455	575	2 120	2 610	1 770	2 180	4 470	5 310	3 730	4 420	7 310	8 430	6 090	7 020	10 490	11 800	8 700	9 830
24	545	690	455	575	2 140	2 650	1 780	2 210	4 560	5 450	3 800	4 540	7 520	8 750	6 270	7 290	10 800	12 300	9 030	10 300
26	545	690	455	575	2 150	2 670	1 790	2 220	4 630	5 580	3 850	4 640	7 700	9 050	6 420	7 530	11 200	12 800	9 320	10 700
28	545	690	455	575	2 160	2 700	1 800	2 250	4 680	5 680	3 900	4 740	7 860	9 280	6 550	7 740	11 500	13 200	9 570	11 100
30	545	690	455	575	2 170	2 710	1 810	2 260	4 730	5 770	3 940	4 810	7 990	9 500	6 660	7 920	11 800	13 700	9 800	11 400
Very great	545	690	455	575	2 180	2 770	1 820	2 310	4 910	6 230	4 096	5 190	8 730	11 100	7 270	9 230	13 600	17 300	11 400	14 400

NOTE.—Ordinary Pipe Laying is pipe laying in accordance with customary good practice in tile-drain construction whereby the underside of the pipe is well bedded on soil for 60 to 90 deg. of the circumference.

First-Class Pipe Laying is pipe laying in accordance with the best customary practice in pipe-sewer construction, whereby the entire underside of the pipe is very thoroughly bedded on soil and the entire pipe is surrounded by well-compacted soil, under the direction of an inspector constantly present on the work.

When pipe is laid in a Concrete or Other Permanent Masonry Cradle, strong enough to carry the entire load to the sub-base without breaking and large enough to prevent material settlement, the standard strengths for all dimensions of ditches and all filling materials shall be those specified for Standard Drain Tile in Table I.

shale and fireclay tile for all other proportions.

Appeal from Absorption

34. In the event that a standard sample (Section 21) of tile fails to meet the requirements of the absorption test, the manufacturer or other seller may demand recourse to the freezing-and-thawing test, to be made at his expense. In such recourse, the number of tiles tested shall be four times the number represented by the standard sample (Section 21). If the material passes the freezing-and-thawing test satisfactorily, it shall not be rejected on account of its failure to meet the absorption requirements specified in Table I, but the average percentage of absorption of the specimens used in the freezing-and-thawing test shall be adopted as the maximum allowable mean absorption for the contract in question.

Limits of Fluctuation of Individual Test Specimens in Physical Tests

35. (*a*) In the strength tests, individual tiles of a standard test whose mean strength is satisfactory may fall 25 per cent below the requirement for the average without causing rejection. In the absorption test, the absorption of individual tiles of a standard sample (Section 21) which gives a satisfactory mean absorption percentage, may exceed the average by 25 per cent without causing rejection. In the freezing-and-thawing test, at least 95 per cent of all the tiles tested shall meet the requirement.

(*b*) In the event of the failure of a standard sample (Sections 9, 21, and 26) to meet the above requirements, the manufacturer or other seller may thoroughly cull the material and submit a portion for retest at his own expense, and for such retest the number of tiles per sample shall be 10 for the strength and absorption tests and 20 for the freezing-and-thawing test. In the event of the failure of the material after culling to pass the requirements, it shall be rejected without further test.

NOTE.–When the freezing-and-thawing test is made as provided in Section 7, the number of freezings and thawings to be endured shall be as follows: For farm drain tile, 24; for standard drain tile, 36; for extra-quality drain tile, 48.

Strength when Manufacturer is held Responsible for Cracking in Ditches

36. The manufacturer or other seller shall not be held responsible for cracking of drain tile in ditches unless by special agreement in advance, and in any event his obligation shall be held to be discharged by the delivery of drain tile having the minimum ordinary supporting strengths specified in Table II; and, if it is not otherwise specified in advance by the purchaser, tile shall be supplied of the strengths specified for clay ditch filling, for "ordinary" pipe laying, and for widths of ditch at the level of the top of the tile equal to 0.5 ft. greater than the outside diameters of the tile. The purchaser shall furnish to the manufacturer or other seller complete information, in advance of receiving bids, as to the number of linear feet of drain tile of each diameter required for each different depth of ditch, measured to the nearest foot from the surface of the ground to the top of the tile.

VISUAL INSPECTION

Visual Inspection

37. All drain tile shall be given a thorough visual inspection at the trench by a competent inspector employed by the purchaser. The purposes of the visual inspection shall be: (1) to cull and reject imperfect individual tiles, and (2) to determine whether the tiles, independently of meeting the chemical and the physical test requirements,

comply with the specifications of general properties, especially as stated hereinafter.

Shape

38. All drain tile shall be of approximately circular cross-section, except when otherwise specified in advance. They shall be approximately straight, except in the case of special connections. The ends shall be so regular and smooth as readily to admit of making close joints by turning and pressing together adjoining tile.

Nominal Sizes

39. The sizes of drain tile shall be designated by their interior diameters.

Minimum Lengths

40. Drain tile smaller than 12 in. in diameter shall have a minimum length of 12 in. Tile of from 12 to 30 in. in diameter, inclusive, shall have lengths not less than the diameters. Tile larger than 30 in. in diameter shall have a minimum length of 30 in.

Structure

41. Drain tile shall be substantially uniform in structure throughout, and the inspector shall investigate this property by examining fractured surfaces.

Ring

42. Drain tile shall give a clear ring when stood on end and while dry tapped with a light hammer.

Color

43. The inspector may use the color of drain tile as a general guide in sorting and inspecting, but he shall first so familiarize himself with the raw materials and the processes used in the manufacture of the particular tile in question as to be competent to interpret the true meaning of variations in their color.

Inside Smoothness

44. Drain tile shall be reasonably smooth on the inside.

Cracks, Checks, Chips, and Broken Pieces

45. Drain tile shall be free from cracks and checks extending into the body of the tile in such a manner as to decrease the strength appreciably. Tile shall not be chipped or broken in such a manner as to decrease their strength materially or to admit earth into the drain.

Use of the Terms Vitrified and Hard Burned

46. All drain tile shall be sufficiently "vitrified" or "hard-burned" to afford the degree of supporting strength, percentage of absorption, and resistance to freezing and thawing specified in the physical test requirements prescribed in Table I.

Appeal from Results of Visual Inspection

47. The manufacturer or other seller may appeal from decisions of the inspector on questions of strength or structure when such decisions are based on visual inspection alone, in which case the point at issue shall be determined by standard physical tests, the cost of which shall be paid by the appellant, if the inspector was right, or by the purchaser if his inspector was in error.

Additional Distinctive Characteristics

48. Drain tile of the different classes shall, in addition to all requirements heretofore specified, have the distinctive physical characteristics prescribed in Table III.

TESTING, INSPECTION, AND REJECTION

Making and Reporting Tests

49. All tests shall be made by experts employed by the purchaser. Full reports of all tests shall be furnished the manufacturer or other seller on his request. Tests shall be made and reported promptly.

TABLE III.—DISTINCTIVE GENERAL PHYSICAL PROPERTIES OF DIFFERENT CLASSES OF DRAIN TILE.

Physical Properties Specified.	Farm Drain Tile.	Standard Drain Tile.	Extra-Quality Drain Tile.
Allowable variation of average diameter below specified diameter, per cent	5	3	3
Allowable variation between maximum and minimum diameters of same tile, or average diameters of adjoining tile, percentage of thickness of wall	85	75	65
Allowable variation from straightness, percentage of length	5	3	3
Allowable thickness of exterior blisters, lumps, and flakes which do not weaken tile and are few in number, percentage of thickness of wall	25	20	15
Allowable diameters of above blisters, lumps, and flakes, percentage of internal diameter	20	15	10
General inspection	careful	rigid	very rigid

Expense of Making Tests

50. The purchaser shall pay the expense of making all tests except as otherwise specified in Sections 9, 34, 35, 47, and 52.

Number of Tests

51. The number of standard tests to be made shall be determined by the purchaser.

General Tests and Inspection at Factory

52. In all contracts for ten or more carloads of tile, preliminary general tests and inspection shall be made at the place of manufacture by the purchaser upon demand of the manufacturer or other seller. The expense of such tests and inspection shall be paid by the manufacturer or other seller.

Inspector

53. The inspector shall be employed by the purchaser.

Inspection

54. The manufacturer or other seller of the drain tile shall afford the inspector all reasonable facilities for his work, both as to the selection of specimens for tests, and as to visual inspection. Inspection shall be completed promptly.

Rejection

55. The inspector shall plainly mark all drain tile which he rejects, and such rejected tile shall be removed promptly by the manufacturer or other seller. Upon request of the purchaser, the manufacturer or other seller shall give full account of the removal of rejected tile.

Standard Specifications for

CONCRETE SEWER PIPE[1]

A.S.T.M. Designation: C 14 - 41

ADOPTED, 1920; REVISED, 1924, 1935, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 14; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover nonreinforced concrete pipe intended to be used for the conveyance of sewage, industrial wastes, and storm water.

Class

2. Pipe manufactured according to these specifications shall be known as "Standard Nonreinforced Concrete Sewer Pipe."

Basis of Acceptance

3. The acceptability of the pipe shall be determined by the results of the strength, absorption, and hydrostatic tests prescribed in these specifications, if and when required, and by inspection to determine whether the pipe conforms to these specifications as to design and freedom from defects.

MATERIALS

Concrete

4. The concrete shall consist of portland cement, mineral aggregates, and water.

Cement

5. Portland cement shall conform to the requirements for type I or type III as prescribed in the Standard Specifications for Portland Cement (A.S.T.M. Designation: C 150) of the American Society for Testing Materials.[3]

Aggregates

6. Aggregates shall conform to the Standard Specifications for Concrete Aggregates (A.S.T.M. Designation: C 33) of the American Society for Testing Materials.[3]

Mixture

7. The aggregates shall be so graded and proportioned and thoroughly mixed

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-13 on Concrete Pipe.

[2] Prior to adoption as standard, these specifications were published as tentative from 1917 to 1920, being revised in 1918, 1919, and 1920.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

in a batch mixer with such proportions of cement and water as will produce a homogeneous concrete mixture of such quality that the pipe will conform to the test and design requirements of these specifications. In no case, however, shall the proportion of portland cement in the mixture be less than six U. S. standard bags (94 lb.) per cubic yard of concrete.

PHYSICAL TEST REQUIREMENTS

Crushing Strength and Absorption

8. The crushing strength and absorption of nonreinforced concrete sewer pipe shall conform to the requirements prescribed in Table I. The individual results of the various tests for each size of pipe and for each shipment and plant shall be tabulated separately so as to show the percentage that fails to conform to the requirements of each test.

TABLE I.—PHYSICAL TEST REQUIREMENTS OF CONCRETE SEWER PIPE.

NOTE.—For pipe 24 to 108 in. in internal diameter, see the Standard Specifications for Reinforced Concrete Sewer Pipe (A.S.T.M. Designation: C 75) of the American Society for Testing Materials.[3]

Internal Diameter, in.	Average Strength, lb. per linear foot		Maximum Absorption, per cent
	Three-Edge-Bearing Method[a]	Sand-Bearing Method[b]	
4	1000	1500	8
6	1100	1650	8
8	1300	1950	8
10	1400	2100	8
12	1500	2250	8
15	1750	2620	8
18	2000	3000	8
21	2200	3300	8
24	2400	3600	8

[a] See Section 15.
[b] See Section 16.

Hydrostatic Test

9. When subjected to the internal hydrostatic pressures specified in Section 19, the pipe shall show no leakage. Moisture appearing on the surface of the pipe in the form of patches or beads adhering to the surface shall not be considered leakage.

Test Specimens

10. (*a*) The specimens to be tested shall be selected by the purchaser or his representative at the point or points designated by him when placing the order.

(*b*) The manufacturer or seller shall furnish, without charge, specimens for tests, up to 0.5 per cent of the number of pipe of each size included in the order, except that in no case shall less than two specimens be furnished. The test specimens shall be sound, full-size pipe.

Measurement and Observation of Specimens

11. (*a*) The test specimens shall first be freed from all visible moisture. When dry, each specimen shall be weighed, measured, and inspected. The results of these observations shall be recorded.

(*b*) *Defective Specimens.*—Specimens which, when placed in a vertical position, do not give a metallic ring when struck with a hammer, or are observed to have cracks or other defects in form or dimensions in excess of the limits permitted in these specifications, shall be discarded and replaced with additional specimens from the shipment.

Crushing Strength Tests

Crushing Strength Tests

12. Pipe shall be tested for crushing strength by either the three-edge-bearing method or the sand-bearing method as described in the following Sections 15 and 16. Either of the two specified bearing methods may be used on retests as provided for in Section 20 (*a*) and (*b*).

Number of Tests

13. The crushing strength test shall ordinarily be applied to not less than

75 per cent of the specimens received for purpose of tests.

Apparatus and Application of Load

14. (*a*) Any prime mover or hand-power device that will apply the load at a uniform rate of about 2000 lb. per min., or in increments of not more than 100 lb. at the same rate, may be used in making the test.

(*b*) The center of the load shall be applied to the top bearing block at a point distant from the spigot end of the pipe equal to one half of the over-all length of the pipe. The test load shall be applied to the upper bearing block in such a way as to leave the bearing free to move in a vertical plane passing midway between the lower bearings. In testing pipe that is "out of straight," the lines of the bearings chosen shall be from those which appear to give most favorable conditions for fair test.

(*c*) The pipe shall not be allowed to stand under load longer than is required to apply the load and to observe and record it.

(*d*) The testing machine shall be substantial and rigid throughout, so that the distribution of the load will not be affected appreciably by the deformation or yielding of any part. The bearings shall be sufficiently rigid to transmit and receive uniform loads throughout their lengths without deflection, and shall be so attached to the machine as to transmit and receive the maximum stresses produced by the tests without lost motion, vibration, or sudden shock.

(*e*) The bearings and the specimen shall be accurately centered so as to secure a symmetrical distribution of the loading on each side of the center of the pipe in every direction.

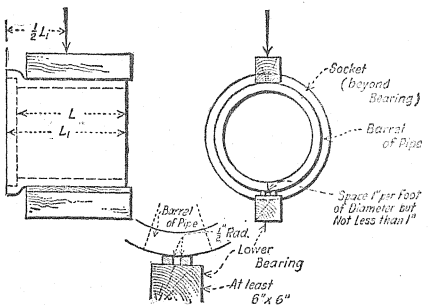


FIG. 1.—Three-Edge Bearings.

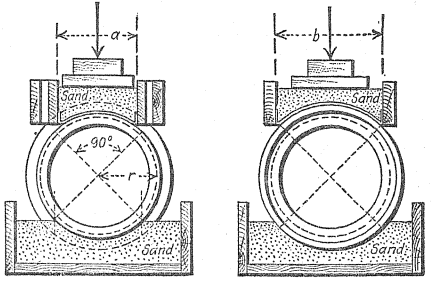
(*f*) The load shall be applied until the pipe yields by cracks passing through the shell.

(*g*) The crushing strength shall be calculated by dividing the total load required to break each pipe by the laying length of the barrel of the pipe, measuring from the bottom of the socket to the end of the spigot.

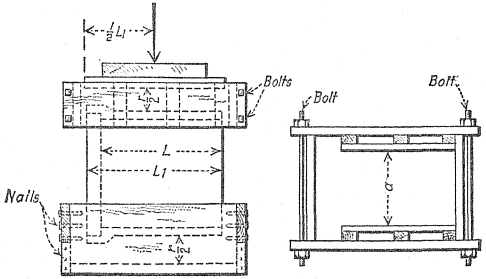
Three-Edge-Bearing Method

15. When the three-edge-bearing method (see Fig. 1) is used, the ends of each specimen of pipe shall be accurately marked in halves of the circumference prior to the test. The lower bearings

shall consist of two wooden strips with vertical sides having their interior top corners rounded to a radius of approximately $\frac{1}{2}$ in. The strips shall be straight and shall be securely fastened to a rigid block at least 6 by 6 in. in cross-section. the pipe is placed, a fillet of plaster of Paris and sand thick enough to compensate for the inequalities of the pipe barrel shall be cast on the surface of the lower bearings. The pipe shall be placed upon the fillet while the plaster



Bedding of Barrel. Bedding of Socket.



Side View. Upper Bearing Frame.

FIG. 2.—Sand Bearings.

The interior vertical sides of the strips shall be parallel and spaced a distance apart of 1 in. per foot of pipe diameter but in no case less than 1 in. If requested by the manufacturer or the purchaser prior to the test, before of Paris is still somewhat plastic. The upper bearing shall be a rigid wooden block at least 6 by 6 in. in cross-section, straight and true from end to end. A fillet of plaster of Paris may also be cast along the length of the crown of the

pipe to equalize the bearing. The upper bearing shall be brought in contact while the plaster of Paris is still somewhat plastic. The upper and lower bearings shall extend the full length of the pipe exclusive of the socket.

Sand-Bearing Method

16. (*a*) When the sand-bearing method (see Fig. 2) is used, the ends of each specimen of pipe shall be accurately marked in quarters of the circumference prior to the test. Specimens shall be carefully bedded in sand, above and below, for one fourth the circumference of the pipe measured on the middle line of the barrel. The depth of the bedding above and below the pipe at the thinnest points shall be one half the radius of the middle line of the barrel.

(*b*) The sand used shall be clean and shall contain not less than 5 per cent of moisture, and shall be such as will pass a 4760-micron (No. 4) sieve. The sand in the lower bearing shall be loose when the pipe is placed.

(*c*) The top bearing frame shall not be allowed to come in contact with the pipe nor with the top bearing plate. The upper surface of the sand in the top bearing shall be struck off level with a straightedge, and shall be covered with a rigid top bearing plate, the lower surface of which is a true plane, made of heavy timbers or other rigid material capable of distributing the test load uniformly without appreciable bending The test load shall be applied at the exact center of this top bearing plate, in such a manner as to permit free motion of the plate in all directions. For this purpose a spherical bearing is preferred, but two rollers at right angles may be used.

(*d*) The test may be made without the use of a testing machine, by piling weights directly on a platform resting on the top bearing plate; provided, however, that the weights shall be piled symmetrically about a vertical line through the center of the pipe, and that the platform shall not be allowed to touch the top bearing frame.

(*e*) The frames of the top and bottom bearings shall be made of timbers so heavy as to avoid appreciable bending by the side pressure of the sand. The interior surfaces of the frames shall be dressed. No frame shall come in contact with the pipe during the test. A strip of cloth may be attached, if desired, to the inside of the upper frame on each side, along the lower edge, to prevent the escape of sand between the frame and the pipe.

Absorption Test

Absorption Test

17. Absorption tests shall be made in accordance with the procedure described in the following Paragraphs (*a*) to (*e*):

(*a*) *Test Specimens.*—The number of absorption test specimens shall be equal to the number of pipe provided for testing. The specimens shall be obtained from pipe that are acceptable as to strength, and shall be taken from pipe used in making the strength test after that test is made. The specimens shall be marked with the number or identification mark of the pipe from which they were taken. Each specimen shall have an area of 12 to 20 sq. in., as measured on one surface of the pipe, and a thickness equal to the full depth of the pipe shell, and shall be free from visible cracks.

(*b*) *Drying Specimens.*—Specimens shall be dried at a temperature not to exceed 230 F. (110 C.) until two successive weighings at intervals of not less than 2 hr. show an increment of loss not greater than 0.1 per cent of the original weight of the specimen.

(*c*) *Immersion and Reweighing.*—The dried specimens shall be placed in a

suitable receptacle, covered with distilled water or rain water, raised to the boiling point and boiled for 5 hr., and then cooled in water to a final temperature of from 59 to 68 F. (15 to 20 C.). When cool, the specimens shall be removed from the water and allowed to drain for not more than 1 min. The superficial water shall then be removed by a towel or blotting paper, and the specimens immediately weighed.

(*d*) *Weighing Apparatus.*—The balance used shall be sensitive to 0.5 g. when loaded with 1 kg., and weighings shall be read at least to the nearest gram. When other than metric weights are used, the same degree of accuracy shall be obtained.

(*e*) *Calculations and Report.*—The increase in weight of the boiled specimen over its dry weight shall be taken as the absorption of the specimen, and shall be expressed as a percentage of the dry weight. The results shall be reported separately for each specimen.

Hydrostatic Test

Number of Tests

18. Sound, full-size pipe not exceeding about 25 per cent of the specimens received for purpose of tests in each size of pipe, shall be tested for leakage under internal hydrostatic pressure.

Hydrostatic Test

19. (*a*) The ends of the pipe shall be tightly closed by wooden or metallic bulkheads or covers faced with rubber or leather so that no leakage shall occur through the covers at the test pressure. One cover shall be provided with a $\frac{3}{4}$-in. nipple passing through the cover, and held securely in place and made watertight by means of locknuts and washers or gaskets. The outer end of the nipple shall be connected with a pump discharge or water service line.

(*b*) Water pressures, as measured by a standardized gage attached to the delivery pipe close to the specimen, shall be internally applied to the specimen as follows:

5 psi. for 5 min.
10 psi. for 10 min.
15 psi. for 15 min.

ACCEPTANCE OR REJECTION ON RESULTS OF TESTS

Acceptance or Rejection

20. (*a*) Failure of 20 per cent of the specimens to meet the requirements of any of the tests specified in Sections 4 to 19 shall result in rejection of all the pipe in the shipment or delivery, corresponding to the sizes thus failing to comply; except that in the event of 20 per cent of the specimens in any size failing to meet the requirements, the manufacturer or seller may, with the consent of the purchaser, furnish for test, without charge, additional specimens from the same shipment to be selected as specified in Section 10. In case more than 80 per cent of the specimens tested, including those first tested, shall show substantial conformance for each of the various tests performed, then the entire shipment or delivery for this size shall be accepted; otherwise it shall be rejected.

(*b*) In addition to the requirements in Paragraph (*a*), failure of individual specimens tested to develop 75 per cent of the average crushing strength requirements shall be cause for rejection of the shipment, but the manufacturer or seller may cull the pipe and submit the balance of the shipment for retest, and if the shipment then conforms to all of the requirements of these specifications it shall be accepted.

SIZES AND PERMISSIBLE VARIATIONS

Sizes and Dimensions

21. Pipe shall be furnished of the sizes, internal diameters, and dimensions prescribed in Tables II and III. Where several lengths are mentioned in the table the purchaser shall indicate, at

the time of purchase, which lengths shall be furnished; and unless so indicated, the manufacturer shall furnish such lengths as he may elect.

TABLE II.—DIMENSIONS OF CONCRETE SEWER PIPE.

NOTE.—For pipe 24 to 108 in. in internal diameter, see the Standard Specifications for Reinforced Concrete Sewer Pipe (A.S.T.M. Designation: C 75) of the American Society for Testing Materials.[3]

Internal Diameter (*D*), in.	Laying Length (*L*), ft.	Inside Diameter of Socket (*D*$_s$), in.[a]	Depth of Socket (*L*$_s$), in.	Minimum Taper of Socket (*H*)	Thickness of Barrel (*T*), in.	Thickness of Socket (*T*$_s$)
4....	2, 2½, 3	6	1½	1:20	⅝	The thickness of the socket ¼ in. from its outer end shall be not less than three fourths of the thickness of the barrel of the pipe.
6....	2, 2½, 3	8¼	2	1:20	⅝	
8....	2, 2½, 3, 4	10¾	2¼	1:20	⅝	
10....	2, 2½, 3, 4	13	2½	1:20	⅞	
12....	2, 2½, 3, 4	15¼	2½	1:20	1	
15....	2, 2½, 3, 4	18¾	2½	1:20	1¼	
18....	2, 2½, 3, 4	22¼	2¾	1:20	1½	
21....	2, 2½, 3, 4	26	2¾	1:20	1¾	
24....	2, 2½, 3, 4	29½	3	1:20	2	

[a] When pipes are furnished having an increase in thickness over that given in last column, then the diameter of socket shall be increased by an amount equal to twice the increase of thickness of barrel.

TABLE III.—PERMISSIBLE VARIATIONS IN DIMENSIONS OF CONCRETE SEWER PIPE.

NOTE.—For pipe 24 to 108 in. in internal diameter, see the Standard Specifications for Reinforced Concrete Sewer Pipe (A.S.T.M. Designation: C 75) of the American Society for Testing Materials.[3]

Nominal Size, Internal Diameter, in.	Limits of Permissible Variation in:				
	Length, in. per ft. (−)[a]	Internal Diameter, in.		Depth of Socket, in. (−)[a]	Thickness of Barrel, in. (−)[a]
		Spigot (±)[a]	Socket (±)[a]		
4.....	1/4	1/16	1/16	1/16	1/16
6.....	1/4	5/64	5/64	1/16	1/16
8.....	1/4	1/16	1/16	1/16	1/16
10.....	1/4	1/16	1/16	1/16	1/16
12.....	1/4	1/16	1/16	1/16	1/16
15.....	1/4	1/16	1/16	1/16	1/16
18.....	1/4	1/16	1/16	1/16	1/16
21.....	1/4	5/64	5/64	1/16	1/16
24.....	5/8	5/64	5/64	1/16	1/16

[a] The minus sign (−) alone indicates that the plus variation is not limited; the plus and minus sign (±) indicates variation in both excess and deficiency in dimension.

Permissible Variations in Dimensions

22. The permissible variations from the dimensions prescribed in Table II shall not exceed those stated in Table III. Where the thickness of barrel is increased beyond that given in Table II in order to meet the specified requirements of strength, the diameter at the inside of the socket shall be increased by double the increase in thickness of the barrel. Pipe intended to be straight shall not have variation in alignment of more than $\frac{1}{8}$ in. per foot of length.

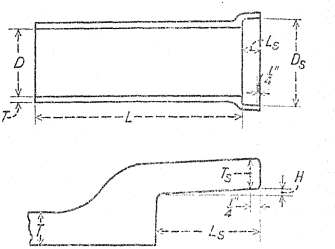


FIG. 3.—Application of Dimensions Given in Table II.

WORKMANSHIP AND FINISH

Absence of Defects

23. Pipes shall be substantially free from fractures, large or deep cracks and blisters, laminations, and surface roughness.

Finish of Ends

24. The ends of the pipes shall be square with their longitudinal axis.

Specials

25. (*a*) Special shapes shall have a plain spigot end and a socket end corresponding in all respects with the dimensions specified for pipe of the corresponding internal diameter. Branches shall be furnished to lay the same lengths as straight pipe. All specials shall conform in finish to the specifications for pipes given in Sections 23 and 24.

(*b*) Slants shall have their spigot ends cut at an angle of approximately 45 deg. with the longitudinal axis.

(*c*) Curves shall be at angles of 90, 45, 22½ deg., as required. They shall con-

form substantially to the curvature specified.

(*d*) Branches shall be furnished with the connection or connections of the size or sizes specified, securely and completely fastened in the process of manufacture to the barrel of the pipe. T-branches and double T-branches shall have their axes perpendicular to the longitudinal axis of the pipe. Y-branches, double Y-branches, and V-branches shall have their axes approximately 45 deg. from the longitudinal axis of the pipe measured from the socket end. All branches shall terminate in sockets, and the barrel of the branch shall be of sufficient length to permit making a proper joint when the connecting pipe is inserted in the branch socket.

(*e*) Channel or split pipe, curves and branches shall be accurate half sections of the corresponding size of pipe or other specials.

MARKING

Marking

26. Each length of pipe shall bear the initials or name of the person, company, or corporation by whom manufactured, and the location of the plant. The markings shall be indented or stenciled on the exterior or interior of the barrel near the socket and shall be plainly legible for purpose of identification.

INSPECTION AND REJECTION

Inspection

27. Each length of pipe shall be subject to inspection at the factory, trench, or other point of delivery by a competent inspector employed by the purchaser. The purposes of the inspection shall be to cull and reject pipe which, independent of the physical tests herein specified, fails to conform to the requirements of these specifications.

Rejection

28. Pipe shall be subject to rejection on account of any of the following:

(*a*) Variations in any dimension exceeding the permissible variations prescribed in Table III.

(*b*) Fractures or cracks passing through the shell or socket, except that a single crack not exceeding 2 in. in length at either end of a pipe or a single fracture in the socket not exceeding 3 in. in width nor 2 in. in length shall not be considered cause for rejection unless these defects exist in more than 5 per cent of the entire shipment or delivery.

(*c*) Blisters where the surface is broken or which project more than $\frac{1}{8}$ in. above the surface.

(*d*) Defects that indicate imperfect mixing and molding.

(*e*) Cracks sufficient to impair the strength, durability, or serviceability of the pipe.

(*f*) Variation of more than $\frac{1}{8}$ in. per linear foot in alignment of pipe intended to be straight.

(*g*) Failure to give a clear ringing sound when placed on end and dry-tapped with a light hammer.

(*h*) Insecure attachment of branches on spurs.

(*i*) The complete absence of distinct web-like markings, which is indicative of a possible deficiency of water in the concrete mix, from the external surface of pipe made by any process in which the forms are removed immediately after the concrete has been placed.

Marking of Rejected Specimens

29. All rejected pipe shall be plainly marked by the inspector and shall be replaced by the manufacturer or seller with pipe which will meet the requirements of these specifications, without additional cost to the purchaser.

Standard Specifications for

REINFORCED CONCRETE SEWER PIPE[1]

A.S.T.M. Designation: C 75 – 41

ADOPTED, 1935; REVISED, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 75; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover reinforced concrete pipe intended to be used for the conveyance of sewage, industrial wastes, and storm water.

Class

2. Pipe manufactured according to these specifications shall be known as "Standard Reinforced Concrete Sewer Pipe."

Basis of Acceptance

3. The acceptability of the pipe shall be determined by the results of the strength and absorption tests prescribed in these specifications, if and when required, and by inspection to determine whether the pipe conforms to these specifications as to design and freedom from defects.

MATERIALS

Reinforced Concrete

4. The reinforced concrete shall consist of portland cement, mineral aggregates, and water, in which steel has been embedded in such a manner that the steel and the concrete act together.

Cement

5. Portland cement shall conform to the requirements for type I or type III as prescribed in the Standard Specifications for Portland Cement (A.S.T.M. Designation: C 150) of the American Society for Testing Materials.[3]

Steel Reinforcement

6. Reinforcement may consist either of wire conforming to the Standard Specifications for Cold-Drawn Steel Wire for Concrete Reinforcement (A.S.T.M. Designation: A 82),[4] or of bars of structural or intermediate grade conforming to the Standard Specifications for Billet-Steel Bars for Concrete Rein-

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-13 on Concrete Pipe.

[2] Prior to adoption as standard, these specifications were published as tentative from 1930 to 1935, being revised in 1934 and 1935.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

[4] 1944 Book of A.S.T.M. Standards, Part I.

forcement (A.S.T.M. Designation: A 15)[4] of the American Society for Testing Materials.

Aggregates

7. Aggregates shall conform to the Standard Specifications for Concrete Aggregates (A.S.T.M. Designation: C 33) of the American Society for Testing Materials.[3]

shall the proportion of portland cement in the mixture be less than six U. S. standard bags (94 lb.) per cubic yard of concrete.

DESIGN

Minimum Designs

9. The shell thickness and the amount of circumferential reinforcement shall

TABLE I.—DESIGN AND STRENGTH TEST REQUIREMENTS OF STANDARD REINFORCED CONCRETE SEWER PIPE.

NOTE.—For pipe 4 to 24 in. in internal diameter, see the Standard Specifications for Concrete Sewer Pipe (A.S.T.M. Designation: C 14) of the American Society for Testing Materials.[3]

Internal Diameter, in.	Strength Test Requirements, lb. per linear ft.				Minimum Design Requirements[a]					
	Three-Edge-Bearing Method		Sand-Bearing Method		Concrete, 3000 psi.		Concrete, 3500 psi.		Concrete, 4000 psi.	
	Load to Produce a 0.01-in. Crack	Ultimate Load	Load to Produce a 0.01-in. Crack	Ultimate Load	Shell Thickness, in.	Total Steel Area, sq. in. per linear ft.	Shell Thickness, in.	Total Steel Area, sq. in. per linear ft.	Shell Thickness, in.	Total Steel Area, sq. in. per linear ft.
12	1800	2700	2700	4050	2	1 line 0.06	1¾	1 line 0.07	....	....
15	2000	3000	3000	4500	2¼	1 line 0.06	2	1 line 0.07	....	....
18	2200	3300	3300	4950	2½	1 line 0.06	....	....	2	1 line 0.07
21	2400	3600	3600	5400	2¾	1 line 0.06	....	....	2¼	1 line 0.07
24	2400	3600	3600	5400	3	1 line 0.06	2⅝	1 line 0.08	2½	1 line 0.09
27	2550	3800	3800	5700	3	1 line 0.07	2⅝	1 line 0.10	2⅝	1 line 0.12
30	2700	4050	4050	6100	3½	1 line 0.09	3	1 line 0.12	2¾	1 line 0.14
33	2850	4300	4300	6400	3¾	1 line 0.11	3½	1 line 0.14	2¾	1 line 0.17
36	3000	4500	4500	6750	4	2 lines[b] totalling 0.14	3⅝	2 lines[b] totalling 0.20	3	2 lines[b] totalling 0.23
42	3200	4800	4800	7200	4½	2 lines[b] totalling 0.16	3⅞	2 lines[b] totalling 0.23	3⅛	2 lines[b] totalling 0.27
48	3400	5100	5100	7650	5	2 lines[b] totalling 0.21	4½	2 lines[b] totalling 0.27	3⅝	2 lines[b] totalling 0.32
54	3700	5550	5550	8300	5½	2 lines[b] totalling 0.25	4⅝	2 lines[b] totalling 0.32	4¼	2 lines[b] totalling 0.38
60	4000	6000	6000	9000	6	2 lines[b] totalling 0.29	5	2 lines[b] totalling 0.38	4½	2 lines[b] totalling 0.44
66	4250	6350	6350	9550	6½	2 lines[b] totalling 0.32	5⅜	2 lines[b] totalling 0.44	4¾	2 lines[b] totalling 0.47
72	4500	6750	6750	10100	7	2 lines[b] totalling 0.36	5¾	2 lines[b] totalling 0.47	5	2 lines[b] totalling 0.55
78	....	....	....	....	7½	2 lines[b] totalling 0.40	....	....	....	....
84	....	....	....	....	8	2 lines[b] totalling 0.43	....	....	....	....
90	....	....	....	....	8	2 lines[b] totalling 0.49	....	....	....	....
96	....	....	....	....	8½	2 lines[b] totalling 0.57	....	....	....	....
108	....	....	....	....	9	2 lines[b] totalling 0.67	....	....	....	....

[a] The distance from the center line of the reinforcement to the nearest surface of the concrete has been assumed in the design tables as 1 in.

[b] Where two lines of steel are specified, a single line placed elliptically may be used, and the area of this shall be at least 50 per cent of the total steel area specified in the design table.

Mixture

8. The aggregates shall be so graded and proportioned and thoroughly mixed in a batch mixer with such proportions of cement and water as will produce a homogeneous concrete mixture of such quality that the pipe will conform to the test and design requirements of these specifications. In no case, however, be not less than the minimum design requirements prescribed in Table I.

Alternate Designs

10. Manufacturers may submit to the purchaser, for approval, designs other than those in Table I, provided that such pipe shall conform to the same

physical tests and inspection requirements prescribed in these specifications for the pipe for which it is substituted. In no alternate design, however, shall the shell thickness be less than that prescribed in Table I.

Placing Reinforcement

11. When two lines of reinforcement are used in circular pipe, one shall be placed near the inner and one near the outer surfaces of the pipe. When a single line of circular reinforcement is used in circular pipe, it shall be placed near the inner surface of the pipe shell. The single line of elliptical reinforcement used in circular pipe, or the single line of circular reinforcement in elliptical pipe shall be placed near the inner surface at the "top" and "bottom" of the pipe and near the outer surface at the sides (see Section 32 (*d*)).

Longitudinals

12. Each line of circumferential reinforcement shall be assembled into a cage which shall contain sufficient longitudinal bars or members, extending through the barrel of the pipe, to maintain the reinforcement rigidly in exact shape and correct position within the form.

Laps, Welds, and Spacing

13. If the splices are not welded, the reinforcement shall be lapped not less than 30 diameters for bars, and 40 diameters for cold-drawn wire. If welded, the member at either a welded splice or intersection shall develop a tensile strength not less than the minimum strength required for the reinforcement by the applicable specifications cited in Section 6. The spacing center to center of adjacent rings of circumferential reinforcement in a cage shall not exceed 4 in. for pipe up to and including 48 in. in diameter, nor exceed the shell thickness for larger pipe, and shall in no case exceed 6 in.

Joints

14. The ends of reinforced concrete sewer pipe shall be so formed that when the pipe are laid together and the joints cemented, they will make a continuous and uniform line of pipe with a smooth and regular interior surface. The joints shall be of such design as will permit effective cementing to reduce leakage and infiltration to a satisfactory minimum and to permit placement without appreciable irregularities in the flow line.

Joint Reinforcement

15. In all pipe 36 in. or more in diameter, the joint shall have a circumferential reinforcement equal in area to that of a single line within the barrel of the pipe.

PHYSICAL TEST REQUIREMENTS

Strength Requirements

16. The ultimate load, as determined by either of the methods described in Sections 23 and 24, shall be not less than the ultimate load prescribed in Table I. When the test load reaches the cracking load given in the table for the size of pipe tested, there shall be in the barrel of the pipe no crack having a width of 0.01 in. or more for a length of 1 ft. or more. The crack shall be considered 0.01 in. in width when the point of the measuring gage will, without forcing, penetrate it $\frac{1}{16}$ in. at close intervals throughout the specified distance of 1 ft. The width of crack shall be measured by means of a gage made from a leaf 0.01 in. in thickness (as in a set of standard machinist's gages), ground to a point $\frac{1}{16}$ in. in width, with corners rounded, and a taper of $\frac{1}{4}$ in. per inch, as illus-

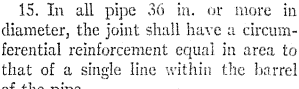


FIG. 1.—Gage Leaf for Measuring Cracks.

trated in Fig. 1. The ultimate load is reached when the pipe will sustain no greater load.

Strength Requirements, Elliptical Pipe

17. Elliptical pipe shall conform to the test requirements for circular pipe having the same horizontal internal diameter.

Test Requirements and Acceptability Under Absorption Tests

18. The absorption, determined as specified in Section 27, shall not exceed 8 per cent of the dry weight. Pipe shall be considered as conforming to these specifications for absorption when not less than 80 per cent of the number of specimens tested, including any retested, conform to the test requirements. When the initial absorption specimen from a pipe fails to conform to these specifications, the absorption test shall be made on another specimen from the same pipe and the results of the retest shall be substituted for the original test results.

Crushing Strength Tests

Test Specimens

19. The specified number of pipe for purpose of tests shall be furnished without charge by the manufacturer and shall be selected at random by the purchaser, and shall be pipe which would not otherwise be rejected under these specifications. The selection shall be made at the point or points designated by the purchaser when placing the order. The test specimens shall be surface-dry when tested and shall not have been exposed to a temperature below 40 F. (5 C.) for the 24 hr. immediately preceding the test.

Preliminary Tests and Tests for Extended Deliveries

20. A purchaser of pipe, whose needs require shipments at intervals over extended periods of time, shall be entitled to preliminary tests of not more than three sections of pipe covering each size, 12 to 72 in. in diameter, in which he is interested. The acceptability of 78- to 108-in. pipe shall be determined by tests of the quality of the concrete as placed in the pipe and by examination of the quality, amount, and accuracy of placement of the reinforcement. The strength of the concrete shall be determined from 6 by 12-in. test cylinders made from the concrete used in making the pipe and manufactured and cured under identical conditions with the pipe. Compression tests of such cylinders shall be made in accordance with the Standard Method of Test for Compressive Strength of Molded Concrete Cylinders (A.S.T.M. Designation: C 39) of the American Society for Testing Materials.[3] After these preliminary tests, a purchaser shall be entitled to additional tests in such numbers and at such times as he may deem necessary, provided that the total number of pipe tested shall not exceed 0.5 per cent of the pipe delivered.

Crushing Strength Tests

21. Pipe may be tested for strength by either the three-edge-bearing method or the sand-bearing method, as described in Sections 23 and 24.

Apparatus

22. (*a*) In making the test, any mechanical or hand-power device may be used in which the head that applies the load moves at such a speed as to increase the load at a uniform rate of approximately 2000 lb. per linear foot of pipe per minute.

(*b*) It is necessary that the testing machine used for the strength tests shall produce a uniform deflection throughout the full length of the pipe. The testing machine shall be substantial and rigid throughout, so that the distribution of

the load will not be affected appreciably by the deformation or yielding of any part.

(*c*) The load shall be applied continuously until the required strength of the pipe specified in Table I is reached. The load per linear foot of pipe shall be calculated by dividing the total recorded load by the laying length. The pipe shall not be allowed to stand under load longer than is required to apply the load and to observe and record it. The pipe shall be surface-dry when tested.

Three-Edge-Bearing Method

23. When the three-edge-bearing method is used (see Figs. 2 and 3), the ends of each specimen of pipe shall be accurately marked in halves of the circumference prior to the test. The lower bearings shall consist of two wooden strips with vertical sides having their interior top corners rounded to a radius of approximately $\frac{1}{2}$ in. The strips shall be straight and shall be securely fastened to a rigid base. The interior vertical

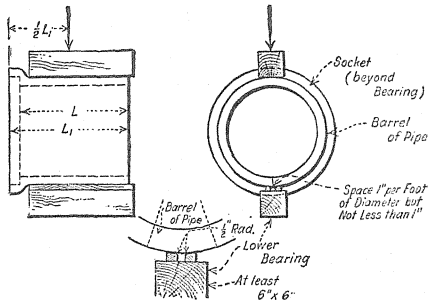


FIG. 2.—Three-Edge Bearings.

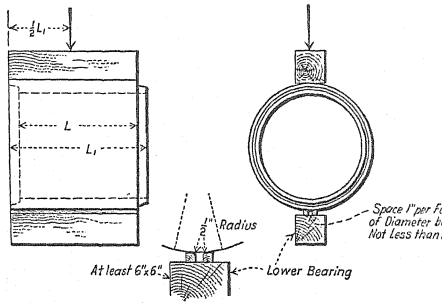


FIG. 3.—Three-Edge Bearings.

sides of the strips shall be parallel and spaced a distance apart of 1 in. per foot of pipe diameter, but in no case less than 1 in. The upper bearing shall be a rigid wooden block, straight and true from end to end. The upper and lower bearings shall extend the full length of the pipe exclusive of the bell. The pipe shall be placed symmetrically between the two bearings as illustrated in Figs. 2 and 3. In testing pipe which is "out of line," the lines of the bearings chosen shall be from those which appear to give the most favorable conditions for fair test.

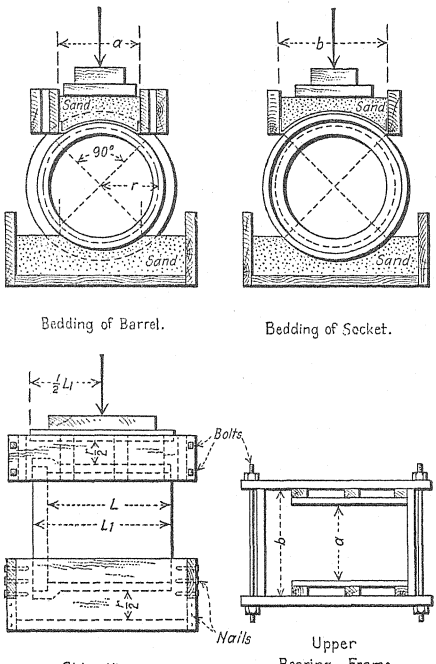


FIG. 4.—Sand Bearings.

Sand-Bearing Method

24. (*a*) When the sand-bearing method is used (see Figs. 4 and 5), the ends of each specimen of pipe shall be accurately marked in quarters of the cir-

cumference prior to the test. Specimens shall be carefully bedded in sand, above and below, for one fourth the circumference of the pipe measured on the middle line of the barrel. The depth of bedding above and below the pipe at the thinnest points shall be one half the radius of the middle line of the barrel.

(*b*) The sand used shall be clean and shall contain not less than 5 per cent of moisture, and shall be such as will pass a 4760-micron (No. 4) sieve. The sand in the lower bearing shall be loose when the pipe is placed.

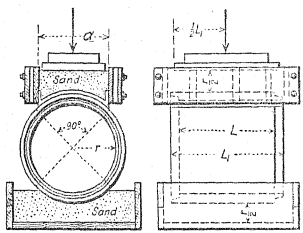


FIG. 5.—Sand Bearings.

(*c*) The top bearing frame shall not be allowed to come in contact with the pipe nor with the top bearing plate. The upper surface of the sand in the top bearing shall be struck off level with a straightedge, and shall be covered with a rigid top bearing plate, the lower surface of which is a true plane, made of heavy timbers or other rigid material capable of distributing the test load uniformly without appreciable bending. The test load shall be applied at the exact center of this top bearing plate, or in such manner as to produce uniform deflection throughout the full length of the pipe. For this purpose a spherical bearing is preferred, but two rollers at right angles may be used.

(*d*) The test may be made without the use of a testing machine, by piling weights directly on a platform resting on the top bearing plate; provided, however, that the weights shall be piled symmetrically about a vertical line through the center of the pipe, and that the platform shall not be allowed to touch the top bearing frame.

(*e*) The frames of the top and bottom bearings shall be made of timbers so heavy as to avoid appreciable bending by the side pressure of the sand. The interior surfaces of the frames shall be dressed. No frame shall come in contact with the pipe during the test. A strip of cloth may be attached, if desired, to the inside of the upper frame on each side, along the lower edge, to prevent the escape of sand between the frame and the pipe.

Acceptability and Retests

25. (*a*) Should the test specimens selected by the purchaser conform to the test requirements, then all pipe represented by such specimens shall be accepted as to strength, provided the pipe shall otherwise conform to the specifications.

(*b*) Should any of the specimens first tested fail to conform to the test requirements, then the manufacturer shall have the right to additional tests of the size or sizes of pipe which have failed. If the strength attained by a specimen is less than the strength required, in the amounts shown in the first column of the following table, additional tests shall be made upon pipe to the number specified below, for each pipe of deficient strength:

Percentage of Required Strength	Number of Pipe for Retest
Less than 100 and more than 90 per cent	2
Less than 90 and more than 80 per cent	3
Less than 80 and more than 70 per cent	4

If the additional pipe so tested all conform to the test requirements, then all pipe represented by such tests shall be accepted; otherwise they shall be rejected, unless other tests, agreed upon by the purchaser and manufacturer, demonstrate to the satisfaction of the purchaser, the acceptability of the pipe. This latter provision, as to tests by agreement, shall also apply in the event that the strength attained by a pipe under test is less than 70 per cent of the required strength.

Concrete Tests and Reinforcement Examination

26. By agreement between the purchaser and the manufacturer, the acceptability of all sizes of pipe may be determined by tests of the quality of concrete as placed in the pipe and the examination of the quality, amount, and accuracy of placement of the reinforcement. The strength of the concrete shall be determined as specified in Section 20.

Absorption Test

Absorption Test

. 27. Absorption tests shall be made in accordance with the procedure described in the following Paragraphs (*a*) to (*e*):

(*a*) *Test Specimens.*—The number of absorption test specimens shall be equal to the number of pipe provided for testing. The specimens shall be obtained from pipe that are acceptable as to strength, and shall be taken from pipe used in making the strength test after that test is made. The specimens shall be marked with the number or identification mark of the pipe from which they were taken. Each specimen shall have an area of 16 to 24 sq. in., as measured on one surface of the pipe, and a thickness equal to the full depth of the pipe shell, and shall be free from visible cracks.

(*b*) *Drying Specimens.*—Specimens shall be dried at a temperature not to exceed 230 F. (110 C.) until two successive weighings at intervals of not less than 2 hr. show an increment of loss not greater than 0.1 per cent of the original weight of the specimen.

(*c*) *Immersion and Reweighing.*—The dried specimens shall be placed in a suitable receptacle, covered with distilled water or rain water, raised to the boiling point and boiled for 5 hr., and then cooled in water to a final temperature of from 59 to 68 F. (15 to 20 C.). When cool, the specimens shall be removed from the water and allowed to drain for not more than 1 min. The superficial water shall then be removed by a towel or blotting paper, and the specimens immediately weighed.

(*d*) *Weighing Apparatus.*—The balance used shall be sensitive to 0.5 g. when loaded with 1 kg., and weighings shall be read at least to the nearest gram. When other than metric weights are used, the same degree of accuracy shall be obtained.

(*e*) *Calculations and Report.*—The increase in weight of the boiled specimen over its dry weight shall be taken as the absorption of the specimen, and shall be expressed as a percentage of the dry weight. The results shall be reported separately for each specimen.

Test Equipment

28. Every manufacturer furnishing pipe under these specifications shall furnish all facilities necessary to carry out the tests required in these specifications.

SIZES AND PERMISSIBLE VARIATIONS

Standard Sizes

29. Pipe of the internal diameters listed in Table I shall be considered

standard sizes. In elliptical pipe, the inside diameter at the minor axis shall be equal to the diameter of the corresponding size of circular pipe.

Permissible Variations in Dimensions

30. (*a*) Variations of the internal diameter shall not exceed 1.5 per cent for 12- to 36-in. pipe, inclusive, and 1 per cent for 42- to 108-in. pipe, inclusive. The shell thickness shall not be less than that given in Table I by more than 5 per cent at any point.

(*b*) Variations of the position of the reinforcement shall not exceed $\frac{1}{4}$ in. from the position provided in the design for pipe having an internal diameter of 48 in. or less, and shall not exceed $\frac{1}{2}$ in. for larger pipe; but the cover on the reinforcement shall be not less than $\frac{3}{4}$ in. at any point.

(*c*) The underrun in length of pipe from that specified shall be not more than $\frac{1}{8}$ in. per ft. with a maximum of $\frac{1}{2}$ in. in any length of pipe.

WORKMANSHIP AND FINISH

Finish

31. Pipe shall be substantially free from fractures, large or deep cracks, and surface roughness. The planes of the ends of the pipe shall be perpendicular to their longitudinal axes.

MARKING

Markings

32. The following shall be clearly stenciled on each length of pipe:

(*a*) The pipe class by an "S" for Standard Pipe,

(*b*) The date of manufacture,

(*c*) The name or trade-mark of the manufacturer, and

(*d*) Elliptical pipe with circular reinforcing and circular pipe with elliptical reinforcing shall have the word "Top" or "Bottom" clearly stenciled on the inside of the pipe at the correct place to indicate the proper position when laid.

INSPECTION AND REJECTION

Age for Acceptance

33. Pipe shall be considered ready for acceptance when they conform to the requirements, as indicated by the specified tests.

Inspection

34. The quality of all materials, the process of manufacture, and the finished pipe shall be subject to inspection and approval by an inspector employed by the purchaser. The manufacturer, when so directed by the inspector, shall have holes cut in such sections of the finished pipe (not exceeding one hole in every 50 sections delivered), as desired, so that a proper inspection may be made of the quantity and placement of the reinforcement. If the pipe is also tested for strength or absorption, inspection of the reinforcement shall be made on the pipe sections used for those tests, and in no case shall the total number of pipe cut open for inspection of reinforcement exceed the number to which the purchaser is entitled under the provisions of Section 20 or 25.

Rejection

35. Pipe shall be subject to rejection on account of failure to conform to any of the specification requirements or on account of any of the following:

(*a*) Fractures or cracks passing through the shell, except that a single end crack that does not exceed the depth of the joint shall not be cause for rejection. If a single end crack that does not exceed the depth of the joint exists in more than 10 per cent of the pipe inspected, however, the defective pipe shall be rejected.

(*b*) Defects that indicate imperfect mixing and molding.

(*c*) Surface defects indicating honeycombed or open texture.

(*d*) Spalls deeper than one half the depth of the joint or extending more than 4 in. around the circumference. If spalls not deeper than one half the depth of the joint or extending not more than 4 in. around the circumference exist in more than 10 per cent of the pipe, however, the defective pipe shall be rejected.

(*e*) Exposure of the circumferential reinforcement when such exposure would indicate that the reinforcement is misplaced.

(*f*) The complete absence of distinct web-like markings, which is indicative of a possible deficiency of water in the concrete mix, from the external surface of pipe made by any process in which the forms are removed immediately after the concrete has been placed.

Standard Specifications for

REINFORCED CONCRETE CULVERT PIPE[1]

A.S.T.M. Designation: C 76 – 41

ADOPTED, 1937; REVISED, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 76; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover reinforced concrete pipe intended to be used for the construction of culverts.

Classes

2. Pipe manufactured according to these specifications shall be of two classes known respectively as "Standard-Strength Reinforced Concrete Culvert Pipe" and "Extra-Strength Reinforced Concrete Culvert Pipe."

Basis of Acceptance

3. The acceptability of the pipe shall be determined by the results of the strength and absorption tests prescribed in these specifications, if and when required, and by inspection to determine whether the pipe conforms to these specifications as to design and freedom from defects.

MATERIALS

Reinforced Concrete

4. The reinforced concrete shall consist of portland cement, mineral aggregates, and water, in which steel has been embedded in such a manner that the steel and the concrete act together.

Cement

5. Portland cement shall conform to the requirements for type I or type III as prescribed in the Standard Specifications for Portland Cement (A.S.T.M. Designation: C 150) of the American Society for Testing Materials.[3]

Steel Reinforcement

6. Reinforcement may consist either of wire conforming to the Standard Specifications for Cold-Drawn Steel Wire for Concrete Reinforcement (A.S.T.M. Designation: A 82),[4] or of bars of structural or intermediate grade conforming to the Standard Specifications

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-13 on Concrete Pipe.

[2] Prior to adoption as standard, these specifications were published as tentative from 1930 to 1937, being revised in 1935 and 1937.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

[4] 1944 Book of A.S.T.M. Standards, Part I.

TABLE I.—DESIGN AND STRENGTH TEST REQUIREMENTS OF STANDARD-STRENGTH REINFORCED CONCRETE CULVERT PIPE.

Internal Diameter of Pipe, in.	Concrete, 3500 psi.			Concrete, 4500 psi.			Strength Test Requirements, lb. per linear ft. of pipe	
	Minimum Reinforcement,[a] sq. in. per linear ft. of pipe barrel			Minimum Reinforcement,[a] sq. in. per linear ft. of pipe barrel			Three-Edge-Bearing Method[b]	
	Minimum Shell Thickness, in.	Circular Reinforcement in Circular Pipe	Elliptical Reinforcement in Circular Pipe and Circular Reinforcement in Elliptical Pipe	Minimum Shell Thickness, in.	Circular Reinforcement in Circular Pipe	Elliptical Reinforcement in Circular Pipe and Circular Reinforcement in Elliptical Pipe	Load to Produce a 0.01-in. Crack	Ultimate Load
12	2	1 line 0.07	.....	1¾	1 line 0.08	.....	2250	3 500
15	2¼	1 line 0.09	.....	2	1 line 0.11	.....	2625	4 065
18	2½	1 line 0.12	1 line 0.10	2	1 line 0.14	.....	3000	4 500
24	3	1 line 0.17	1 line 0.13	2½	1 line 0.20	1 line 0.17	3000	5 000
30	3½	1 line 0.22	1 line 0.17	3	1 line 0.28	1 line 0.21	3375	5 750
36	4	2 lines, each 0.18	1 line 0.18	3½	2 lines, each 0.22	1 line 0.22	4050	6 600
42	4½	2 lines, each 0.21	1 line 0.21	3½	2 lines, each 0.25	1 line 0.25	4725	7 350
48	5	2 lines, each 0.25	1 line 0.25	4¼	2 lines, each 0.31	1 line 0.31	5400	8 000
54	5½	2 lines, each 0.30	1 line 0.30	4½	2 lines, each 0.37	1 line 0.37	5850	9 000
60	6	2 lines, each 0.33	1 line 0.33	5	2 lines, each 0.41	1 line 0.41	6000	10 000
66	6½	2 lines, each 0.37	1 line 0.37	5½	2 lines, each 0.45	1 line 0.45	6300	11 000
72	7	2 lines, each 0.40	1 line 0.40	6	2 lines, each 0.48	1 line 0.48	6600	12 000
78	7½	2 lines, each 0.43	1 line 0.43	6½	2 lines, each 0.51	1 line 0.51	....	....
84	8	2 lines, each 0.46	1 line 0.46	7	2 lines, each 0.54	1 line 0.54	....	....
90	8	2 lines, each 0.56	1 line 0.56	....	.....	.....	....	....
96	8½	2 lines, each 0.60	1 line 0.60	....	.....	.....	....	....
102	8½	2 lines, each 0.72	1 line 0.72	....	.....	.....	....	....
108	9	2 lines, each 0.78	1 line 0.78	....	.....	.....	....	....

[a] The distance from the center line of the reinforcement to the nearest surface of the concrete has been assumed in th design tables as 1¼ in. for pipe with a shell 2½ in. or more in thickness.
[b] Test loads for sand-bearing tests shall be one and one-half times those specified in this table for the three-edge-bearing tests.

TABLE II.—DESIGN AND STRENGTH TEST REQUIREMENTS OF EXTRA-STRENGTH REINFORCED CONCRETE CULVERT PIPE.

Internal Diameter of Pipe, in.	Concrete, 4500 psi.			Strength Test Requirements, lb. per linear ft. of pipe	
	Minimum Shell Thickness, in.	Minimum Reinforcement,[a] sq. in. per linear ft. of pipe barrel		Three-Edge-Bearing Method[b]	
		Circular Reinforcement in Circular Pipe	Elliptical Reinforcement in Circular Pipe and Circular Reinforcement in Elliptical Pipe	Load to Produce a 0.01-in. Crack	Ultimate Load
24	3	1 line 0.26	1 line 0.20	4 000	6 000
30	3½	1 line 0.31	1 line 0.24	5 000	7 500
36	4	2 lines, each 0.28	1 line 0.28	6 000	9 000
42	4½	2 lines, each 0.33	1 line 0.33	7 000	10 500
48	5	2 lines, each 0.38	1 line 0.38	8 000	12 000
54	5½	2 lines, each 0.44	1 line 0.44	9 000	13 500
60	6	2 lines, each 0.50	1 line 0.50	9 000	15 000
66	6½	2 lines, each 0.56	1 line 0.56	9 500	16 500
72	7	2 lines, each 0.60	1 line 0.60	9 900	18 000
78	7½	2 lines, each 0.65	1 line 0.65	....	....
84	8	2 lines, each 0.72	1 line 0.72	....	....
90	8	2 lines, each 0.84	1 line 0.84	....	....
96	8½	2 lines, each 0.90	1 line 0.90	....	....
102	8½	2 lines, each 1.08	1 line 1.08	....	....
108	9	2 lines, each 1.17	1 line 1.17	....	....

[a] The distance from the center line of the reinforcement to the nearest surface of the concrete has been assumed in the design tables as 1¼ in. for pipe with a shell 2½ in. or more in thickness.
[b] Test loads for sand-bearing tests shall be one and one-half times those specified in this table for the three-edge-bearing tests.

for Billet-Steel Bars for Concrete Reinforcement (A.S.T.M. Designation: A 15)[4] of the American Society for Testing Materials.

Aggregates

7. Aggregates shall conform to the Standard Specifications for Concrete Aggregates (A.S.T.M. Designation: C 33) of the American Society for Testing Materials.[3]

Mixture

8. The aggregates shall be so graded and proportioned and thoroughly mixed in a batch mixer with such proportions of cement and water as will produce a homogeneous concrete mixture of such quality that the pipe will conform to the test and design requirements of these specifications. In no case, however, shall the proportion of portland cement in the mixture be less than six U. S. standard bags (94 lb.) per cubic yard of concrete.

DESIGN

Minimum Designs

9. The shell thickness and the amount of circumferential reinforcement shall be not less than that prescribed in Tables I and II.

Alternate Designs

10. Manufacturers may submit to the purchaser, for approval, designs other than those in Tables I and II, provided that such pipe shall conform to the same physical tests and inspection requirements prescribed in these specifications for the pipe for which it is substituted. In no alternate design, however, shall the shell thickness be less than that prescribed in Tables I and II.

Placing Reinforcement

11. In circular pipe having one line of circular reinforcement, the reinforcement in the barrel of the pipe shall be placed equally distant from the inner and outer surfaces of the pipe. In circular pipe having two lines of circular reinforcement, each line shall be so placed that the net protective covering of concrete over the reinforcement in the barrel of the pipe shall be not less than 1 in. In circular pipe having elliptical reinforcement, and in elliptical pipe having circular reinforcement, the reinforcement in the barrel of the pipe shall be so placed that the net protective covering of concrete along the vertical diameter of the pipe shall be not less than 1 in. from the inside surface of the pipe, and the net protective covering of concrete along the horizontal diameter of the pipe shall be not less than 1 in. from the outside surface of the pipe. (See Section 34 (*d*)). The location of the reinforcement shall be subject, however, to the variation permitted in Section 32 (*b*).

Longitudinals

12. Each line of circumferential reinforcement shall be assembled into a cage which shall contain sufficient longitudinal bars or members, extending through the barrel of the pipe, to maintain the reinforcement rigidly in exact shape and correct position within the form.

Laps, Welds, and Spacing

13. If the splices are not welded, the reinforcement shall be lapped not less than 30 diameters for bars, and 40 diameters for cold-drawn wire. If welded, the member at either a welded splice or intersection shall develop a tensile strength not less than the minimum strength required for the reinforcement by the applicable specifications cited in Section 6. The spacing center to center of adjacent rings of circumferential

reinforcement in a cage shall not exceed 4 in. for pipe up to and including 48 in. in diameter, nor exceed the shell thickness for larger pipe, and shall in no case exceed 6 in.

Joints

14. The ends of reinforced concrete culvert pipe shall be of such design that the pipe when laid shall form a continuous conduit with a smooth and uniform interior surface.

Joint Reinforcement

15. In all pipe 36 in. or more in diameter, the joint shall have a circumferential reinforcement equal in area to that of a single line within the barrel of the pipe.

CURING

Curing

16. Pipe shall be subjected to any one of the methods of curing described in the following Paragraphs (*a*) to (*c*) or to any other method or combination of methods, approved by the purchaser, that will give satisfactory results:

(*a*) *Steam Curing.*—Pipe may be placed in a curing chamber, free from outside drafts, and cured in a moist atmosphere, maintained at a temperature between 100 and 130 F. (38 and 54 C.) by the injection of steam for a period of not less than 24 hr. or, when necessary, for such additional time as may be needed to enable the pipe to meet the strength requirements. When a curing chamber is not available, pipe may be placed in an enclosure of canvas or other closely woven material and subjected to saturated steam at the temperature and for the time specified above. The enclosure shall be so erected as to allow full circulation of steam around the entire pipe. The interior surfaces of the curing room or canvas jackets and the surfaces of the pipe shall be entirely moist at all times.

(*b*) *Water Spray Curing.*—Under the conditions of enclosure prescribed in Paragraph (*a*), pipe may be cured by subjecting it to a continuous or frequently applied fine spray of water in an enclosure maintained at a temperature of not less than 70 F. (21 C.) for a period of not less than 72 hr., or such additional time as may be necessary to meet the strength requirements. If the enclosure is maintained at a temperature of less than 70 F. (21 C.), the curing period shall be increased as may be necessary to meet the strength requirements.

(*c*) *Saturated Cover Curing.*—The sides and top of each pipe may be covered with heavy burlap or other suitable material, saturated with water before applying and kept saturated with water at a temperature of not less than 70 F. (21 C.) for 72 hr., or such additional time as may be necessary to meet the strength requirements. The ends of the pipe shall be so enclosed as to prevent the free circulation of air through or around the pipe. If the temperature of the water is less than 70 F. (21 C.), the curing period shall be increased as may be necessary to meet the strength requirements.

PHYSICAL TEST REQUIREMENTS

Strength Requirements

17. The ultimate load, as determined by either of the methods described in Sections 25 and 26, shall be not less than the ultimate load prescribed in Tables I and II. When the test load reaches the cracking load given in the tables for the size and class of pipe

tested, there shall be in the barrel of the pipe no crack having a width of 0.01 in. or more for a length of 1 ft. or more. The crack shall be considered 0.01 in. in width when the point of the measuring gage will, without forcing, penetrate it $\frac{1}{16}$ in. at close intervals throughout the specified distance of 1 ft. The width of crack shall be measured by means of a gage made from a leaf 0.01 in. in thickness (as in a set of standard machinist's gages), ground to a point $\frac{1}{16}$ in. in width, with corners rounded, and a taper of $\frac{1}{4}$ in. per inch, as illustrated in Fig. 1. The ultimate load is reached when the pipe will sustain no greater load.

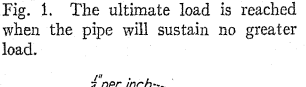


FIG. 1.—Gage Leaf for Measuring Cracks.

Strength Requirements, Elliptical Pipe

18. Elliptical pipe shall conform to the test requirements for circular pipe having the same horizontal internal diameter.

Test Requirements and Acceptability Under Absorption Tests

19. The absorption, determined as specified in Section 29, shall not exceed 8 per cent of the dry weight. Pipe shall be considered as conforming to these specifications for absorption when not less than 80 per cent of the number of specimens tested, including any retested, conform to the test requirements. When the initial absorption specimen from a pipe fails to conform to these specifications, the absorption test shall be made on another specimen from the same pipe and the results of the retest shall be substituted for the original test results.

Crushing Strength Tests

Test Specimens

20. The specified number of pipe for purpose of tests shall be furnished without charge by the manufacturer and shall be selected at random by the purchaser, and shall be pipe which would not otherwise be rejected under these specifications. The selection shall be made at the point or points designated by the purchaser when placing the order. The test specimens shall be surface-dry when tested and shall not have been exposed to a temperature below 40 F. (5 C.) for the 24 hr. immediately preceding the test.

Preliminary Tests and Tests for Extended Deliveries

21. A purchaser of pipe, whose needs require shipments at intervals over extended periods of time, shall be entitled to preliminary tests of not more than three sections of pipe covering each size, 12 to 72 in. in diameter, in which he is interested. The acceptability of 78- to 108-in. pipe shall be determined by tests of the quality of the concrete as placed in the pipe and by examination of the quality, amount, and accuracy of placement of the reinforcement. The strength of the concrete shall be determined from 6 by 12-in. test cylinders made from the concrete used in making the pipe and manufactured and cured under identical conditions with the pipe. Compression tests of such cylinders shall be made in accordance with the Standard Method of Test for Compressive Strength of Molded Concrete Cylinders (A.S.T.M. Designation: C 39) of the American Society for Testing Materials.[3] After these preliminary tests, a purchaser shall be entitled to additional tests in such num-

bers and at such times as he may deem necessary, provided that the total number of pipe tested shall not exceed 2 per cent[5] of the pipe delivered.

an order, and not to exceed five pieces of any one size; otherwise the number of pipe desired for testing shall be included in the order.

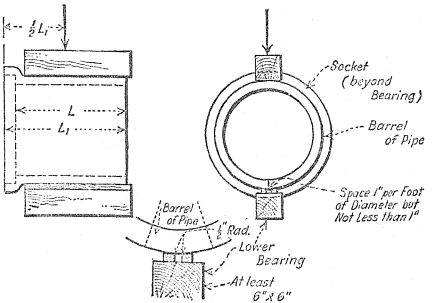


FIG. 2.—Three-Edge Bearings.

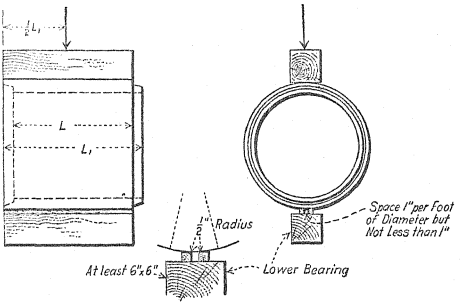


FIG. 3.—Three-Edge Bearings.

Tests for Occasional Orders

22. A purchaser who places occasional orders shall be entitled to test a number of pipe not to exceed 2 per cent[5] of an order, and not to exceed five pieces of any one size; otherwise the number of pipe desired for testing shall be included in the order.

[5] If these specifications are used for sewers, the number of pieces of pipe supplied for testing shall be in accordance with Section 20 of the Standard Specifications for Reinforced Concrete Sewer Pipe (A.S.T.M. Designation: C 75), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Crushing Strength Tests

23. Pipe may be tested for strength by either the three-edge-bearing method or the sand-bearing method, as described in Sections 25 and 26.

Apparatus

24. (*a*) In making the test, any me-

chanical or hand-power device may be used in which the head that applies the load moves at such a speed as to increase the load at a uniform rate of approximately 2000 lb. per linear foot of pipe per minute.

(*b*) It is necessary that the testing machine used for the strength tests shall produce a uniform deflection throughout the full length of the pipe. The testing machine shall be substantial and rigid throughout, so that the distribution of the load will not be affected appreciably by the deformation or yielding of any part.

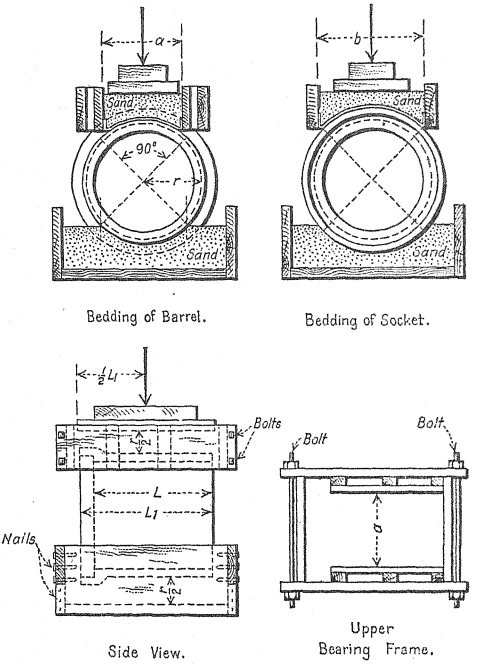


FIG. 4.—Sand Bearings.

(*c*) The load shall be applied continuously until the required strength of the pipe specified in Tables I and II is reached. The load per linear foot of pipe shall be calculated by dividing the total recorded load by the laying length. The pipe shall not be allowed to stand under

load longer than is required to apply the load and to observe and record it. The pipe shall be surface-dry when tested.

Three-Edge-Bearing Method

25. When the three-edge-bearing method is used (see Figs. 2 and 3), the ends of each specimen of pipe shall be accurately marked in halves of the circumference prior to the test. The lower bearings shall consist of two wooden strips with vertical sides having their interior top corners rounded to a radius of approximately $\frac{1}{2}$ in. The strips shall be straight and shall be securely fastened to a rigid base. The interior vertical sides of the strips shall be parallel and spaced a distance apart of 1 in. per foot of pipe diameter, but in no case less than 1 in. The upper bearing shall be a rigid wooden block, straight and true from end to end. The upper and lower bearings shall extend the full length of the pipe exclusive of the bell. The pipe shall be placed symmetrically between the two bearings as illustrated in Figs. 2 and 3. In testing pipe which is "out of line", the lines of the bearings chosen shall be from those which appear to give the most favorable conditions for fair test.

Sand-Bearing Method

26. (*a*) When the sand-bearing method is used (see Figs. 4 and 5), the ends of each specimen of pipe shall be accurately marked in quarters of the circumference prior to the test. Specimens shall be carefully bedded in sand, above and below, for one fourth the circumference of the pipe measured on the middle line of the barrel. The depth of bedding above and below the pipe at the thinnest points shall be one half the radius of the middle line of the barrel.

(*b*) The sand used shall be clean and shall contain not less than 5 per cent of moisture, and shall be such as will pass a 4760-micron (No. 4) sieve. The sand in the lower bearing shall be loose when the pipe is placed.

(*c*) The top bearing frame shall not be allowed to come in contact with the pipe nor with the top bearing plate. The upper surface of the sand in the top bearing shall be struck off level with a straightedge, and shall be covered with a rigid top bearing plate, the lower surface of which is a true plane, made of heavy timbers or other rigid material capable of distributing the test load uniformly without appreciable bending. The test load shall be applied at the exact center of this top bearing plate, or in such manner as to produce uniform deflection throughout the full length of the pipe. For this purpose a spherical bearing is preferred, but two rollers at right angles may be used.

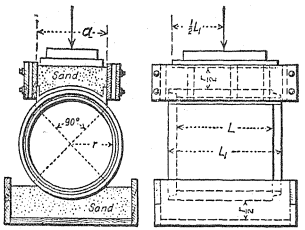


FIG. 5.—Sand Bearings.

(*d*) The test may be made without the use of a testing machine, by piling weights directly on a platform resting on the top bearing plate; provided, however, that the weights shall be piled symmetrically about a vertical line through the center of the pipe, and that the platform shall not be allowed to touch the top bearing frame.

(*e*) The frames of the top and bottom bearings shall be made of timbers so

heavy as to avoid appreciable bending by the side pressure of the sand. The interior surfaces of the frames shall be dressed. No frame shall come in contact with the pipe during the test. A strip of cloth may be attached, if desired, to the inside of the upper frame on each side, along the lower edge, to prevent the escape of sand between the frame and the pipe.

Acceptability and Retests

27. Pipe shall be acceptable under the strength tests when all test specimens conform to the test requirements. Should any of the preliminary test specimens provided for in Section 21, or any of the test specimens provided for in Section 22, fail to meet the test requirements, then the manufacturer will be allowed a retest on two additional specimens for each specimen that failed, and the pipe shall be acceptable only when all of these retest specimens meet the strength requirements.

Concrete Tests and Reinforcement Examination

28. By agreement between the purchaser and the manufacturer, the acceptability of all sizes of pipe may be determined by tests of the quality of concrete as placed in the pipe and the examination of the quality, amount, and accuracy of placement of the reinforcement. The strength of the concrete shall be determined as specified in Section 21.

Absorption Test

Absorption Test

29. Absorption tests shall be made in accordance with the procedure described in the following Paragraphs (*a*) to (*e*):

(*a*) *Test Specimens.*—The number of absorption test specimens shall be equal to the number of pipe provided for testing. The specimens shall be obtained from pipe that are acceptable as to strength, and shall be taken from pipe used in making the strength test after that test is made. The specimens shall be marked with the number or identification mark of the pipe from which they were taken. Each specimen shall have an area of 16 to 24 sq. in., as measured on one surface of the pipe, and a thickness equal to the full depth of the pipe shell, and shall be free from visible cracks.

(*b*) *Drying Specimens.*—Specimens shall be dried at a temperature not to exceed 230 F. (110 C.) until two successive weighings at intervals of not less than 2 hr. show an increment of loss not greater than 0.1 per cent of the original weight of the specimen.

(*c*) *Immersion and Reweighing.*—The dried specimens shall be placed in a suitable receptacle, covered with distilled water or rain water, raised to the boiling point and boiled for 5 hr., and then cooled in water to a final temperature of from 59 to 68 F. (15 to 20 C.). When cool, the specimens shall be removed from the water and allowed to drain for not more than 1 min. The superficial water shall then be removed by a towel or blotting paper, and the specimens immediately weighed.

(*d*) *Weighing Apparatus.*—The balance used shall be sensitive to 0.5 g. when loaded with 1 kg., and weighings shall be read at least to the nearest gram. When other than metric weights are used, the same degree of accuracy shall be obtained.

(*e*) *Calculations and Report.*—The increase in weight of the boiled specimen over its dry weight shall be taken as the absorption of the specimen, and shall be expressed as a percentage of the dry weight. The results shall be reported separately for each specimen.

Test Equipment

30. Every manufacturer furnishing pipe under these specifications shall furnish all facilities necessary to carry out the tests required in these specifications.

SIZES AND PERMISSIBLE VARIATIONS

Standard Sizes

31. Pipe of the internal diameters listed in Tables I and II shall be the standard sizes for culvert construction. In elliptical pipe, the inside diameter at the minor axis shall be equal to the iameter of the corresponding size of circular pipe.

Permissible Variations in Dimensions

32. (*a*) Variations of the internal diameter shall not exceed plus or minus 1 per cent for pipe having an internal diameter of 36 in. or less, and shall not exceed plus or minus 0.75 per cent for larger pipe. The shell thickness shall not be less than that intended in the design by more than 5 per cent at any point.

(*b*) Variations of the position of the reinforcement shall not exceed $\frac{1}{4}$ in. from the position provided in the design for pipe having an internal diameter of 48 in. or less, and shall not exceed $\frac{1}{2}$ in. for larger pipe; but the cover on the reinforcement shall not be less than $\frac{3}{4}$ in. at any point.

(*c*) The underrun in length of pipe from that specified shall be not more than $\frac{1}{8}$ in. per ft. with a maximum of $\frac{1}{2}$ in. in any length of pipe.

WORKMANSHIP AND FINISH

Finish

33. Pipe shall be substantially free from fractures, large or deep cracks, and surface roughness. The planes of the ends of the pipe shall be perpendicular to the longitudinal axis.

MARKING

Marking

34. The following shall be clearly stenciled on the pipe:

(*a*) The pipe class { by a "C" for Standard-Strength Pipe, and / by an "X-C" for Extra-Strength Pipe,

(*b*) The date of manufacture,

(*c*) The name or trade-mark of the manufacturer, and

(*d*) Elliptical pipe with circular reinforcing and circular pipe with elliptical reinforcing shall have the word "Top" or "Bottom" clearly stenciled on the inside of the pipe at the correct place to indicate the proper position when laid.

INSPECTION AND REJECTION

Age for Acceptance

35. Pipe shall be considered ready for acceptance when they conform to the requirements, as indicated by the specified tests.

Inspection

36. The quality of all materials, the process of manufacture, and the finished pipe shall be subject to inspection and approval by an inspector employed by the purchaser. The manufacturer, when so directed by the inspector, shall have holes cut in such sections of the finished pipe (not exceeding one hole in every 50 sections delivered), as desired, so that a proper inspection may be made of the quantity and placement of the reinforcement. If the pipe is also tested for strength or absorption, inspection of the reinforcement shall be made on the pipe sections used for those tests, and in no case shall the total number of pipe cut open for inspection of reinforcement exceed the number to which the purchaser is entitled under the provisions of Section 21, 22, or 27.

Rejection

37. Pipe shall be subject to rejection on account of failure to conform to any of the specification requirements or on account of any of the following:

(*a*) Fractures or cracks passing through the shell, except that a single end crack that does not exceed the depth of the joint shall not be cause for rejection. If a single end crack that does not exceed the depth of the joint exists in more than 10 per cent of the pipe inspected, however, the defective pipe shall be rejected.

(*b*) Defects that indicate imperfect mixing and molding.

(*c*) Surface defects indicating honeycombed or open texture.

(*d*) Spalls deeper than one half the depth of the joint or extending more than 4 in. around the circumference. If spalls not deeper than one half the depth of the joint or extending not more than 4 in. around the circumference exist in more than 10 per cent of the pipe, however, the defective pipe shall be rejected.

(*e*) Exposure of the circumferential reinforcement when such exposure would indicate that the reinforcement is misplaced.

(*f*) The complete absence of distinct web-like markings, which is indicative of a possible deficiency of water in the concrete mix, from the external surface of pipe made by any process in which the forms are removed immediately after the concrete has been placed.

Standard Specifications for
CONCRETE IRRIGATION PIPE[1]

A.S.T.M. Designation: C 118 – 39

ADOPTED, 1939.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 118; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover concrete pipe intended to be used for the conveyance of irrigation water under low hydrostatic heads.

Class

2. Pipe manufactured according to these specifications shall be known as "A.S.T.M. Standard Concrete Irrigation Pipe."

Basis of Acceptance

3. The acceptability of the pipe shall be determined by the results of the tests prescribed in these specifications, if and when required, and by inspection to determine whether the pipe conforms to these specifications as to design and freedom from defects.

MATERIALS

Concrete

4. The concrete shall consist of portland cement, mineral aggregates and water.

Cement

5. Portland cement shall conform to requirements for type I or type III as prescribed in the Standard Specifications for Portland Cement (A.S.T.M. Designation: C 150) of the American Society for Testing Materials.[3]

Aggregates

6. Aggregates shall conform to the Standard Specifications for Concrete Aggregates (A.S.T.M. Designation: C 33) of the American Society for Testing Materials.[3] The maximum size of coarse aggregate used shall not exceed one-third the shell thickness of the pipe.

Mixture

7. The aggregates shall be so graded and proportioned and thoroughly mixed with such proportions of cement and water as will produce a homogeneous concrete mixture of such quality that the pipe will conform to the test and design requirements of these specifications.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-13 on Concrete Pipe.

[2] Prior to adoption as standard, these specifications were published as tentative from 1935 to 1939, being revised in 1938.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

DESIGN AND PERMISSIBLE VARIATIONS IN DIMENSIONS

Alternate Designs

8. Manufacturers may submit to the purchaser, for approval, designs other than those in Table I. In no alternate design, however, shall the shell thickness and test requirements be less than those prescribed in Table I.

Variation in Diameter

9. Variation of the internal diameter shall not exceed plus or minus $\frac{3}{16}$ in. for 6-in. pipe, plus or minus $\frac{1}{4}$ in. for pipe having an internal diameter greater than 6 in. but not more than 18 in., and plus or minus $\frac{5}{16}$ in. for larger pipe.

TABLE I.—PHYSICAL TEST REQUIREMENTS FOR STANDARD CONCRETE IRRIGATION PIPE.

Internal Diameter, in.	Minimum Shell Thickness, in.	Test Requirements		
		Internal Hydrostatic Pressure on Individual Sections, psi.	Minimum Three-Edge-Bearing Load, lb. per linear ft.	Maximum Absorption, per cent
*6	$\frac{3}{4}$	75	1000	8
8	$\frac{7}{8}$	65	1000	8
10	1	60	1250	8
12	$1\frac{1}{8}$	55	1500	8
*14	$1\frac{1}{4}$	50	1600	8
15	$1\frac{3}{8}$	50	1700	8
*16	$1\frac{3}{8}$	50	1800	8
18	$1\frac{1}{2}$	50	1900	8
*20	$1\frac{5}{8}$	45	2000	8
21	2	45	2100	8
24	$2\frac{1}{8}$	40	2200	8

* Special sizes obtainable in some localities.

Variation in Thickness

10. The shell thickness at any point shall not be less than the minimum specified in Table I by more than $\frac{1}{16}$ in. for pipe having an internal diameter of 12 in. or less, $\frac{3}{32}$ in. for pipe having an internal diameter greater than 12 in. but not more than 18 in., and $\frac{1}{8}$ in. for larger pipe.

Joints

11. The ends of concrete irrigation pipe shall be so formed that when the pipe are laid together and the joints cemented they will make a continuous and uniform line of pipe with a smooth and regular interior surface. The joints shall be of such design as will permit effective cementing and placement without appreciable irregularities in the flow line.

PHYSICAL PROPERTIES AND TESTS

Test Specimens

12. (*a*) Specimens for tests shall be full-size pipe which shall in every respect conform to the inspection requirements prescribed in these specifications.

(*b*) The specimens to be tested shall be selected at random by the purchaser or his representative at the point or points specified in the order. The manufacturer shall furnish specimens for purpose of tests, without charge, up to 0.5 per cent of the number of pipe of each size included in the order, except that in no case shall less than two specimens be furnished, the manufacturer bearing all expense of testing such pipe. Should a larger number of specimens be tested upon demand of the purchaser or manufacturer, then the cost of such additional test specimens and the expense of testing shall be borne by the party making such demand.

(*c*) The purchaser shall specify the proportion of the test specimens that shall be subjected to the strength tests and the proportion that shall be subjected to the hydrostatic test.

(*d*) Should the test specimens selected by the purchaser conform to the test requirements, then all pipe represented by such specimens shall be accepted,

provided the pipe shall otherwise conform to the specifications.

(*e*) Should any of the specimens first tested fail to conform to the test requirements, then the manufacturer shall have the right to test two additional lengths of pipe from the same lot, selected by mutual agreement with the purchaser, for each specimen that has failed. If the additional pipe so tested all conform to the test requirements, then all pipe represented by such tests shall be accepted, otherwise they shall be subject to rejection.

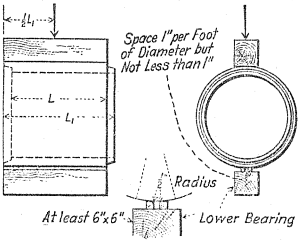


FIG. 1.—Three-Edge Bearings.

Strength Tests

13. Pipe shall be tested for strength by either the three-edge-bearing method or the sand-bearing method, as described in the following Paragraphs (*a*) to (*c*):

Three-Edge-Bearing Method

(*a*) When tested by the three-edge-bearing method (see Fig. 1), individual pipe shall meet the minimum load requirements specified in Table I.

In making the test the ends of each specimen of pipe shall be accurately marked in halves of the circumference prior to the test. The lower bearings shall consist of two wooden strips with vertical sides, having their interior top corners rounded to a radius of approximately $\frac{1}{2}$ in. The strips shall be straight and shall be securely fastened to a rigid block at least 6 by 6 in. in cross-section. The interior vertical sides of the strips shall be parallel and spaced a distance apart of 1 in. per foot of pipe diameter, but in no case less than 1 in. If requested by the manufacturer or the purchaser previous to the test, before the pipe is placed, a fillet of plaster of Paris and sand, thick enough to compensate for the inequalities of the pipe barrel, shall be cast on and between the lower bearings. The pipe shall be placed upon the fillet while the plaster of Paris is still somewhat plastic. The

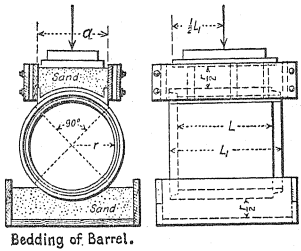


FIG. 2.—Sand Bearings.

upper bearing shall be a rigid wooden block at least 6 by 6 in. in cross-section, straight and true from end to end. A fillet of plaster of Paris may also be cast along the length of the crown of the pipe to equalize the bearing. The upper bearing shall be brought in contact while the plaster of Paris is still somewhat plastic. The upper and lower bearings shall extend the full length of the pipe (see Fig 1).

Sand-Bearing Method

(*b*) When tested by the sand-bearing method (see Fig. 2) individual pipe shall meet a minimum load 50 per cent greater

than specified in Table I for the three-edge-bearing test.

When the sand-bearing method (see Fig. 2) is used the ends of each specimen of pipe shall be accurately marked in quarters of the circumference prior to the test. Specimens shall be carefully bedded in sand, above and below, for one-fourth the circumference of the pipe measured on the middle line of the barrel. The depth of the bedding above and below the pipe at the thinnest points shall be one-half the radius of the middle line of the barrel.

The sand used shall be clean and shall contain not less than 5 per cent of moisture, and shall be such as will pass a 4760-micron (No. 4) sieve. The sand in the lower bearing shall be loose when the pipe is placed.

The top bearing frame shall not be allowed to come in contact with the pipe nor with the top bearing plate. The upper surface of the sand in the top bearing shall be struck off level with a straightedge, and shall be covered with a rigid top-bearing plate, the lower surface of which is a true plane, made of heavy timbers or other rigid material capable of distributing the test load uniformly without appreciable bending. The test load shall be applied at the exact center of this top bearing plate, or in such manner as to produce uniform deflection throughout the full length of the pipe. For this purpose a spherical bearing is preferred, but two rollers at right angles may be used.

The test may be made without the use of a testing machine, by piling weights directly on a platform resting on the top bearing plate; provided, however, that the weights shall be piled symmetrically about a vertical line through the center of the pipe, and that the platform shall not be allowed to touch the top bearing frame.

The frames of the top and bottom bearings shall be made of timbers so heavy as to avoid appreciable bending by the side pressure of sand. The interior surfaces of the frames shall be dressed. No frame shall come in contact with the pipe during the test. A strip of cloth may be attached, if desired, to the inside of the upper frame on each side, along the lower edge, to prevent the escape of sand between the frame and the pipe.

Testing Apparatus

(*c*) In making the test, any mechanical or hand-power device may be used in which the head that applies the load moves at such a speed as to increase the load at a uniform rate of approximately 2000 lb. per linear foot of pipe per minute.

It is necessary that the testing machine used for the strength tests shall produce a uniform deflection throughout the full length of the pipe. The testing machine shall be substantial and rigid throughout, so that the distribution of the load will not be affected appreciably by the deformation or yielding of any part.

The load shall be applied continuously until the required strength of the pipe specified in Table I is reached. The load per linear foot of pipe shall be calculated by dividing the total recorded load by the laying length. The pipe shall not be allowed to stand under load longer than is required to apply the load and to observe and record it. The pipe shall be surface-dry when tested.

Hydrostatic Test

14. (*a*) When an individual pipe is subjected to the internal hydrostatic pressure given in Table I, there shall be no leakage through the shell of the pipe. Moisture appearing on the surface of the pipe in the form of patches or beads

adhering to the surface shall not be considered leakage.

(*b*) The equipment for making the hydrostatic test shall be mutually satisfactory to the purchaser and the manufacturer. Suitable fittings shall be provided for exhausting the air and admitting the water into the specimen. A standardized pressure gage for recording the internal pressure shall be connected close to the specimen. The pressure shall first be increased uniformly to 10 psi. and held at this pressure for 10 min. The water pressure shall then be increased uniformly until the required maximum pressure is reached as specified in Table I. If the pipe is to be coated on the inside with cement grout or other material, the hydrostatic test shall be made before such coating is applied.

Absorption Test

15. Absorption tests shall be made in accordance with the procedure described in the following Paragraphs (*a*) to (*e*):

(*a*) *Test Specimens.*—The number of absorption test specimens shall be equal to the number of pipe provided for testing. The specimens shall be obtained from pipe that are acceptable as to strength, and shall be taken from pipe used in making the strength test after that test is made. The specimens shall be marked with the number or identification mark of the pipe from which they were taken. Each specimen shall have an area of 12 to 20 sq. in., as measured on one surface of the pipe, and a thickness equal to the full depth of the pipe shell, and shall be free from visible cracks.

(*b*) *Drying Specimens.*—Specimens shall be dried at a temperature not to exceed 230 F. (110 C.) until two successive weighings at intervals of not less than 2 hr. show an increment of loss not greater than 0.1 per cent of the original weight of the specimen.

(*c*) *Immersion and Reweighing.*—The dried specimens shall be placed in a suitable receptacle covered with distilled water or rain water, raised to the boiling point and boiled for 5 hr., and then cooled in water to a final temperature of from 59 to 68 F. (15 to 20 C.). When cool, the specimens shall be removed from the water and allowed to drain for not more than 1 min. The superficial water shall then be removed by a towel or blotting paper, and the specimens immediately weighed.

(*d*) *Weighing Apparatus.*—The balance used shall be sensitive to 0.5 g. when loaded with 1 kg., and weighings shall be read at least to the nearest gram. When other than metric weights are used, the same degree of accuracy shall be obtained.

(*e*) *Calculations and Report.*—The increase in weight of the boiled specimen over its dry weight shall be taken as the absorption of the specimen, and shall be expressed as a percentage of the dry weight. The results shall be reported separately for each specimen.

WORKMANSHIP AND FINISH

Finish

16. Pipe shall be concentric, and free from fractures, large or deep cracks and surface roughness, other than those normally produced in the manufacture of the pipe. The planes of the ends of the pipe shall be perpendicular to the longitudinal axis.

INSPECTION AND REJECTION

Age for Shipment

17. Pipe shall be considered ready for acceptance when they conform to the test requirements prescribed in these specifications, and have been properly cured for at least 7 days.

Inspection

18. The quality of all materials and the finished pipe shall be subject to inspection and approval by an inspector employed by the purchaser.

Rejection

19. Pipe shall be subject to rejection on account of failure to conform to any of the specification requirements or on account of any of the following:

(*a*) Fractures or cracks passing through the shell, except that an end crack that does not exceed the depth of the joint, or a fracture that at its deepest point does not exceed the depth of the joint nor extend more than 10 per cent around the circumference of the joint shall not be considered cause for rejection unless these defects exist in more than 5 per cent of the pipe inspected. If these defects exist in more than 5 per cent of the pipe inspected, the defective pipe shall be rejected.

(*b*) Defects which indicate imperfect mixing, placing, and curing of concrete.

(*c*) The absence of distinct web-like markings on the external surface of pipe made by the packerhead or machine-tamped process.

Recommended Practice For

LAYING SEWER PIPE[1]

A.S.T.M. Designation: C 12 – 19

ADOPTED, 1919.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 12; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Preparation of Trenches for Pipe Laying

1. (*a*) Pipe lines should be placed at a sufficient depth below the surface of the street to avoid dangerous pressure or impact. When this is not possible, special reinforcement should be provided.

(*b*) Trenches should be only of sufficient width to provide a free working space on each side of the pipe, preferably of not over one-third of the nominal diameter and never less than 4 in., according to the size of the pipe and the character of the ground; but in every case there should be sufficient space between the pipe and the sides of the trench to make it possible to thoroughly ram the back-filling around the pipe and to secure tight joints.

(*c*) Trenches should be kept free from water until the material in the joints and masonry has sufficiently hardened.

(*d*) To protect pipe lines from unusual stresses, all work should preferably be done in open trenches.

Preparation of Foundations for Pipe Laying

2. (*a*) The foundations in the trench should be formed to prevent any subsequent settlement which might result in excessive pressure and consequent rupture of the pipes.

(*b*) If the foundation is rock, an equalizing bed of concrete or sand well compacted should be placed upon the rock. The thickness of these beds should be not less than 4 in. Pipes should be laid in these beds so that at least the lower third of each pipe is supported throughout its entire length.

(*c*) If the foundation is good firm earth, the earth should be pared or molded to give a full support to the lower third of each pipe and, if necessary to secure a proper bearing for the pipe, a layer of concrete, fine gravel, or other suitable material should be placed. The same means of securing a firm foundation should be adopted in case the excavation has been made deeper than necessary.

[1] Under the standardization procedure of the Society, this recommended practice was formerly under the jurisdiction of the Committee on Clay and Concrete Pipe. In 1932 that committee was divided into two committees, C-4 on Clay Pipe and C-13 on Concrete Pipe, and in October, 1933, this recommended practice was placed under the jurisdiction of Committee C-4 with the understanding that it would be suitably revised to apply to clay pipe only.

[2] Prior to adoption as standard, this recommended practice was published as tentative from 1915 to 1919, being revised in 1916 and 1917.

(*d*) If there is no good natural foundation, the pipes should be laid in a concrete cradle supported on a masonry foundation carried to a soil of satisfactory bearing power, or supported on a structure designed to carry the weight of pipe and its load to a firm bearing.

Pipe Laying

3. (*a*) The laying of pipes in finished trenches should be commenced at the lowest point, so that the spigot ends point in the direction of flow.

(*b*) All pipes should be laid with ends abutting and true to line and grade. They should be fitted and matched so that when laid in the work they will form a sewer with a smooth and uniform invert. It is necessary to use all possible care when shoving the pipes together, so that the joints will not be unnecessarily large.

(*c*) Sockets should be carefully cleaned before pipes are lowered into trenches. The pipes should be so lowered as to avoid unnecessary handling in the trench.

(*d*) The pipes should be set firmly according to line and grade, and the joints carefully adjusted and filled with the jointing material.

(*e*) Joints should be made in the following manner: A closely twisted hemp or oakum gasket of suitable diameter, in no case less than $\frac{3}{4}$ in., and in one piece of sufficient length to pass around the pipe and lap at the top, should be solidly rammed into the annular spaces between the pipes with a suitable calking tool. When cement joints are used, the gasket should first be saturated with neat cement grout. The remainder of the space should then be completely filled with the jointing materials.

Backfilling Trenches

4. (*a*) All trenches and excavations should be backfilled immediately after the pipes are laid therein, unless other protection of the pipe line is directed. The backfilling material should be selected and deposited with special reference to the future safety of the pipes. Clean earth, sand, or rock dust should be solidly tamped about the pipes up to a level at least 2 ft. above the top of the pipes. This material should be carefully deposited in uniform layers. Unless otherwise permitted, each layer should be carefully and solidly tamped or rammed with proper tools so as not to injure or disturb the pipe line.

(*b*) Puddling or water flooding for consolidating the backfilling is recommended only for sandy and gravely materials. If this method is used, the first flooding should be applied after the backfilling has been compacted by tamping up to 2 ft. above the top of the pipes, and the second flooding during or after the subsequent filling of the trench. An excess of water should be avoided, in order to prevent disturbance of the earth under and around the pipes and also to prevent an undue excess of pressure upon them.

(*c*) Walking or working on the completed pipe line, except as may be necessary in tamping or backfilling, should not be permitted until the trench has been backfilled to a height of at least 2 ft. over the top of the pipes.

(*d*) The filling of the trench should be carried on simultaneously on both sides of the pipes, in such a manner that injurious side pressures do not occur.

Standard Specifications for

CONCRETE AGGREGATES[1]

A.S.T.M. Designation: C 33 – 44

ADOPTED, 1939; REVISED, 1940, 1942, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 33; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover fine and coarse aggregates suitable for use in concrete.

NOTE: *Quality of Aggregates.*—It is recognized that for certain purposes satisfactory results may be obtained with materials not conforming to these specifications. In such cases the use of fine and coarse aggregates not conforming to these specifications may be authorized only under special provisions based upon laboratory studies of the possibility of designing a mixture of materials to be used on the job that will yield concrete equivalent in quality to the specified mixture made with material complying with these specifications in all respects.

FINE AGGREGATE

General Characteristics

2. Fine aggregate shall consist of natural sand or of sand prepared from stone, blast-furnace slag, or gravel, or, subject to the approval of the engineer, other inert materials having similar characteristics.

Deleterious Substances

3. (*a*) The amount of deleterious substances in fine aggregate shall not exceed the limits prescribed in Table I.

TABLE I.—PERMISSIBLE LIMITS FOR DELETERIOUS SUBSTANCES IN CONCRETE AGGREGATES.

	Recommended Permissible Limits, max., per cent by weight	Maximum Permissible Limits, per cent by weight
Clay lumps	1	1.5
Coal and lignite	0.25	1
Material finer than No. 200 sieve:		
(*a*) In concrete subject to surface abrasion	2	4
(*b*) All other classes of concrete	3	5
Other deleterious substances (such as shale, alkali, mica, coated grains, soft and flaky particles)	as specified	as specified

NOTE.—The recommended limits should be specified on all work where it is economically practicable to obtain materials conforming thereto.

(*b*) *Organic Impurities.*—All fine aggregate shall be free from injurious amounts of organic impurities. Aggregates subjected to the colorimetric test for organic impurities and producing a color darker than the standard shall be

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates.

[2] Prior to adoption as standard, these specifications were published as tentative from 1921 to 1939, being revised in 1923, 1926, 1928, 1930, 1931, 1936, and 1937.

rejected unless they pass the mortar strength test as specified in Section 5.

Grading

4. (*a*) Fine aggregate shall be well graded from coarse to fine and when tested by means of laboratory sieves shall conform to the following requirements:

Sieve[3]	Percentage Passing
⅜-in.	100
No. 4 (4760-micron)	95 to 100
No. 16 (1190-micron)	45 to 80
No. 50 (297-micron)	10 to 30
No. 100 (149-micron)	2 to 10

NOTE 1.—At the approval of the engineer, when the fine aggregate is to be used in concrete mixtures containing five or more sacks of cement per cubic yard, the limitations on the material passing the No. 50 and No. 100 sieves may be 5 to 30 and 0 to 10 per cent, respectively.

NOTE 2.—Fine aggregate failing to pass the minimum requirements for the material passing the No. 50 or No. 100 sieve, or both, may be used, provided a satisfactory inorganic fine material is added to correct for the difference in grading.

NOTE 3.—Attention is called to the fact that the relatively wide range in grading which is shown in the above table should be permitted only when it is economically impracticable to obtain materials meeting more restrictive requirements. The most desirable grading will depend upon the type of work and the class of concrete. For the leaner mixes, or when a small size coarse aggregate is used, in cases where the degree of workability is important, it is desirable to further restrict the allowable ranges in sizes shown so as to insure a grading approaching the maximum percentage passing each sieve. On the other hand, for the richer mixes, in the interests of maximum strength and economy, a grading as coarse as is consistent with the requirements for workability should be specified. However, in no case should a range in grading be required more restrictive on any one sieve than indicated below:

No. 16 (1190-micron) sieve	20 per cent
No. 50 (297-micron) sieve	15 per cent
No. 100 (149-micron) sieve	5 per cent

(*b*) *Uniformity of Grading.*—The above gradation for fine aggregate represents the extreme limits which shall determine the suitability for use of fine aggregate from all sources of supply. The gradation of fine aggregate from any one source shall be reasonably uniform and not subject to the extreme percentages of gradation specified above. For the purpose of determining the degree of uniformity of a fine aggregate, a fineness modulus determination shall be made upon representative samples of fine aggregate from such sources as are proposed for use. Fine aggregate from any one source having a variation in fineness modulus greater than plus or minus 0.20 from the fineness modulus of the representative sample submitted by the contractor shall either be rejected or may be accepted subject to such adjustment in proportions as may be necessary by reason of changes in grading of fine aggregate. Fine aggregate from different sources of supply shall not be mixed or stored in the same pile nor used alternately in the same class of construction or mix, without permission from the engineer.

(*c*) In case the concrete resulting from a mixture of aggregates approaching the extreme limits for gradation is not of a workable character or when finished does not exhibit a proper surface, due to an excess of particles approximately ⅜ to ½ in. in size, either a fine aggregate having a sufficiently greater percentage of fine material, or a coarse aggregate having a sufficiently smaller percentage of fine material shall be used.

Mortar Strength

5. Fine aggregate shall be of such quality that when made into a mortar and subjected to the mortar strength test (Section 11 (*e*)) it shall develop a compressive strength at 7 and 28 days of not less than .... per cent[4] of that developed by a mortar prepared in the same manner with the same cement and graded standard sand having a fineness modulus of 2.40 ± 0.10.

[3] Detailed requirements for these sieves are given in the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

[4] A percentage should be inserted by the engineer to suit local conditions.

NOTE.—The graded standard sand mentioned in Section 5 may be obtained with a mixture of approximately equal parts by weight of standard Ottawa sand conforming to the requirements prescribed in the Standard Method of Test for Tensile Strength of Hydraulic-Cement Mortars (A.S.T.M. Designation: C 190)[5] and graded Ottawa sand conforming to the requirements specified in Section 4 of the Standard Method of Test for Compressive Strength of Hydraulic-Cement Mortars (A. S. T. M. Designation: C 109).[5]

Soundness

6. Fine aggregate shall pass a sodium or magnesium sulfate accelerated soundness test, except that aggregates failing in the accelerated soundness test may be used if they pass a satisfactory freezing-and-thawing test.

COARSE AGGREGATE

General Characteristics

7. Coarse aggregate shall consist of crushed stone, gravel, blast-furnace slag, or other approved inert materials of similar characteristics, or combinations thereof, having hard, strong, durable pieces, free from adherent coatings and conforming to the requirements of these specifications.

Deleterious Substances

8. The amount of deleterious substances in coarse aggregate shall not exceed the following limits:

	Recommended[a] Permissible Limits, max., per cent by weight	Maximum Permissible Limits, per cent by weight
Soft fragments	2	5
Coal and lignite	0.25	1
Clay lumps	0.25	0.25
Material finer than No. 200 (74-micron) sieve	0.5[b]	1[b]
Other deleterious substances	as specified	as specified

[a] The recommended requirements should be specified on all work where it is economically practicable to obtain materials conforming thereto.

[b] When the material finer than the No. 200 sieve consists essentially of crusher dust, the recommended and maximum permissible limits specified above may be raised to 0.75 and 1.5 per cent, respectively.

[5] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Grading

9. (*a*) Coarse aggregate shall be well graded between the limits specified and shall conform to the requirements prescribed in Table II.

(*b*) Designation of a given coarse aggregate as aggregate of a certain maximum size shall be understood to mean that more than 5 per cent must be retained on the next smaller of the sieves appearing in Table II.

(*c*) In case the concrete resulting from a mixture of aggregates approaching the extreme limits for gradation is not a workable character or when finished does not exhibit a proper surface, due to an excess of particles approximately ¼ to ½ in. in size, either a fine aggregate having a sufficiently greater percentage of fine material or a coarse aggregate having a sufficiently smaller percentage of fine material shall be used.

Weight of Slag

10. Blast-furnace slag that meets the grading requirements of these specifications shall conform to the following weight requirements:

	Compact Weight, min., lb. per cu. ft.
General concrete	65
Concrete subject to abrasion	70

Soundness

11. Coarse aggregate shall pass a sodium or magnesium sulfate accelerated soundness test, except that aggregates failing in the accelerated soundness test may be used if they pass a satisfactory freezing-and-thawing test.

NOTE.—Many engineers believe that an abrasion test for coarse aggregate to be used in concrete subject to abrasion is important, but no test limits are specified, due to the status of knowledge concerning suitable specification limits for this test. The committee believes that the abrasion tests when applied to blast-furnace slag do not meet the requirements for a desirable test. This recommendation is made after consideration of the results of a study to

determine the value of the abrasion test as to the concrete making properties of slag.

Methods of Sampling and Testing

12. The aggregates shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials (except as specified in Paragraph (*j*)):

(*a*) *Sampling.*—Tentative Methods of Sampling Stone, Slag, Gravel, Sand, and Stone Block for Use as Highway Materials (A.S.T.M. Designation: D 75).[5]

(*f*) *Compressive Strength.*—Standard Method of Test for Compressive Strength of Molded Concrete Cylinders (A.S.T.M. Designation: C 39).[5]

(*g*) *Soundness.*—Tentative Method of Test for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (A.S.T.M. Designation: C 88).[5]

(*h*) *Clay Lumps.*—Standard Method of Test for Clay Lumps in Aggregates (A.S.T.M. Designation: C 142).[5]

(*i*) *Coal and Lignite.*—Standard Method of Test for Coal and Lignite in Sand (A.S.T.M. Designation: C 123).[5]

(*j*) *Shale.*—Method of Test for Per-

TABLE II.—GRADING REQUIREMENTS FOR CRUSHED STONE, GRAVEL, AND BLAST-FURNACE SLAG.

Designated Size	Percentages Passing Laboratory Sieves Having Square Openings[a]							
	2½ in.	2 in.	1½ in.	1 in.	¾ in.	½ in.	⅜ in.	No. 4 (4760-micron)
2 in. to No. 4	100	95 to 100	.....	35 to 70	.....	10 to 30	.....	0 to 5
1½ in. to No. 4	...	100	95 to 100	.....	35 to 70	.....	10 to 30	0 to 5
1 in. to No. 4	...	.....	100	90 to 100	.....	25 to 60	.....	0 to 10
¾ in. to No. 4	...	.....	.....	100	90 to 100	.....	20 to 55	0 to 10
½ in. to No. 4[6]	...	.....	.....	.....	100	90 to 100	.....	0 to 15
2 in. to 1 in.	100	90 to 100	35 to 70	0 to 15	.....	.....	.....	.....
1½ in. to ¾ in.	...	100	90 to 100	20 to 55	0 to 15	.....	.....	.....

[a] Not more than 5 per cent passing the No. 8 (2380-micron) sieve.

(*b*) *Sieve Analysis.* — Standard Method of Test for Sieve Analysis of Fine and Coarse Aggregates (A.S.T.M. Designation: C 136).[5]

(*c*) *Amount of Material Finer than No. 200 Sieve.*—Standard Method of Test for Amount of Material Finer than No. 200 Sieve in Aggregates (A.S.T.M. Designation: C 117).[5]

(*d*) *Organic Impurities.*—Standard Method of Test for Organic Impurities in Sands for Concrete (A.S.T.M. Designation: C 40).[5]

(*e*) *Mortar Strength.* — Standard Method of Test for Structural Strength of Fine Aggregate Using Constant Water-Cement-Ratio Mortar (A.S.T.M. Designation: C 87).[5]

centage of Shale in Aggregate,[6] Standard Method T-10 of The American Association of State Highway Officials.

(*k*) *Moisture.*—Tentative Method of Test for Surface Moisture in Fine Aggregate (A.S.T.M. Designation: C 70).[5]

(*l*) *Weight of Slag.*—Standard Method of Test for Unit Weight of Aggregate (A.S.T.M. Designation: C 29).[5]

(*m*) *Abrasion.*—If abrasion tests are made the following methods of test are recommended:

(1) *Abrasion of Gravel.*—Tentative Method of Test for Abrasion of Gravel by Use of the Deval Machine (A.S.T.M. Designation: D 289).[5]

(2) *Abrasion of Rock.*—Standard Meth-

[6] See "Standard Specifications for Highway Materials and Methods of Sampling and Testing," the Am. Assn. State Highway Officials, Part II, p. 201 (1942).

od of Test for Abrasion of Rock by Use of the Deval Machine (A.S. T.M. Designation: D 2),[5] except that for material having specific gravities lower than 2.2 a 4000-g. sample shall be used.

(*n*) *Fineness Modulus.*—The fineness modulus is the sum of percentages in the sieve analysis divided by 100 when the sieve analysis is expressed as cumulative percentages coarser than each of the following sieves: No. 100 (149-micron), No. 50 (297-micron), No. 30 (590-micron), No. 16 (1190-micron), No. 8 (2380-micron), and No. 4 (4760-micron). The sieve analysis shall be made in accordance with Standard Method C 136.[5]

Standard Specifications for

LIGHTWEIGHT AGGREGATES FOR CONCRETE[1]

A.S.T.M. Designation: C 130 - 42

ADOPTED, 1939; REVISED, 1942.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 130; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover lightweight aggregates suitable for use in concrete.

NOTE.—The specification limits that are not enclosed in parentheses are recommended for use when it is practicable to secure economically materials conforming to them. When it is not practicable to secure such materials economically the specification limits may be extended, but not beyond the maximum permissible limits shown in parentheses. Generally, such extension in the specification limits can be compensated for by a change in the design of the mix or by the exercise of more than the usual care in the control of the concrete during construction.

General Characteristics

2. (*a*) Lightweight aggregates shall consist of pumice, lava, tufa, slag, burned clay, burned shale, cinders derived from the high-temperature combustion of coal or coke showing a loss on ignition of not more than 25 per cent (40 per cent)[3] and volatile matter of not more than 5 per cent, or, subject to approval by the engineer, other material having strong, durable particles and conforming to the requirements of these specifications.

(*b*) When approved by the engineer, for the purpose of securing the desired workability, lightweight aggregate meeting the requirements of these specifications may be mixed with a fine aggregate meeting the requirements of the Standard Specifications for Concrete Aggregates (A.S.T.M. Designation: C 33) of the American Society for Testing Materials,[4] provided, however, that the combined aggregate shall conform to all the other requirements of these specifications.

Deleterious Substances

3. (*a*) Lightweight aggregate shall not contain excessive amounts of deleterious substances.

(*b*) *Organic Impurities.*—All lightweight aggregate shall be free from injurious amounts of organic impurities. Aggregates subjected to the colorimetric

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates.

[2] Prior to adoption as standard, these specifications were published as tentative from 1937 to 1939.

[3] See Note under Section 1.

[4] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

test for organic impurities and producing a color darker than the standard shall be rejected unless they pass the mortar strength test (Section 6), which requirement may be raised by the engineer by not more than 10 per cent.

Grading

4. Lightweight aggregate shall be graded from coarse to fine within the limits prescribed in Table I.

Unit Weight

5. Lightweight aggregate meeting the grading requirements of these specifications when tested in a dry condition shall conform to the following weight requirements:

	Unit Weight, max., lb. per cu. ft.
Fine aggregate	75
Coarse aggregate	55

Mortar Strength

6. (*a*) Lightweight aggregate shall be of such quality that when made into a mortar and subjected to the mortar strength test (Section 8 (*d*)) it shall develop a compressive strength at 7 or 28 days of not less than 70 per cent (or, for concrete not exposed to wear or weather, 60 per cent) of that developed by a mortar prepared in the same manner with the same cement and the graded standard sand described in Paragraph (*b*). Coarse aggregate shall be reduced to the grading specified for fine aggregate before use in the mortar test.

(*b*) The standard sand (Note) referred to in Paragraph (*a*) shall be natural silica sand from Ottawa, Ill., graded as follows:

Sieves[5]	Percentage Retained
No. 100 (149-micron)	98 ± 2
No. 50 (297-micron)	72 ± 5
No. 30 (590-micron)	2 ± 2
No. 16 (1190-micron)	none

NOTE.—Sand conforming to the above requirements may be obtained from the Ottawa Silica Co., Ottawa, Ill.

TABLE I.—GRADING REQUIREMENTS FOR LIGHTWEIGHT AGGREGATES.

Size Designation	Percentages Passing Sieves Having Square Openings[5]								
	1 in.	¾ in.	½ in.	⅜ in.	No. 4 (4760 micron)	No. 8 (2380 micron)	No. 16 (1190 micron)	No. 50 (297 micron)	No. 100 (149 micron)
Fine Aggregate:									
¼ in. to dust	....	.....	.....	100	95 to 100	.....	45 to 80	10 to 30	5 to 15
⅜ in. to dust	....	.....	100	95 to 100	55 to 80	.....	.....	10 to 25	5 to 15
Coarse Aggregate:									
½ in. to No. 4	....	100	90 to 100	40 to 75	0 to 15	0 to 5	.....	.....	.....
½ in. to No. 8	....	.....	100	85 to 100	0 to 20	0 to 5	.....	.....	.....
¾ in. to No. 4	100	90 to 100	.....	20 to 55	0 to 10	0 to 5	.....	.....	.....

Soundness

7. Lightweight aggregate, when subjected to five cycles of the accelerated sulfate soundness test, shall lose not more than 12 per cent (15 per cent)[8] in weight, provided, however, that an aggregate failing in this requirement may be accepted if it passes a satisfactory freezing-and-thawing test. The engineer may waive the soundness test requirement for lightweight aggregate for concrete not to be exposed to moisture.

Methods of Sampling and Testing

8. Lightweight aggregate shall be sampled and the properties enumerated

[5] Detailed requirements for these sieves are given in the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(*a*) *Sampling.*—Tentative Methods of Sampling Stone, Slag, Gravel, Sand, and Stone Block for Use as Highway Materials (A.S.T.M. Designation: D 75).[4]

(*b*) *Grading.*—Standard Method of Test for Sieve Analysis of Fine and Coarse Aggregates (A.S.T.M. Designation: C 136).[4]

(*c*) *Unit Weight.*—Standard Method of Test for Unit Weight of Aggregate (A.S.T.M. Designation: C 29),[4] utilizing the jigging procedure described in Section 6 of Methods C 29. Unit weights shall be determined on material dried to constant weights at not less than 100 C. and not more than 120 C.

(*d*) *Mortar Strength.*—Standard Method of Test for Structural Strength of Fine Aggregate Using Constant Water-Cement-Ratio Mortar (A.S.T.M. Designation: C 87).[4]

(*e*) *Compressive Strength.*—Standard Method of Test for Compressive Strength of Molded Concrete Cylinders (A.S.T.M. Designation: C 39).[4]

(*f*) *Soundness.*—Tentative Method of Test for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (A.S.T.M. Designation: C 88).[4]

(*g*) *Organic Impurities.*—Standard Method of Test for Organic Impurities in Sands for Concrete (A.S.T.M. Designation: C 40).[4]

APPROVED AS
AMERICAN STANDARD
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A.A.S.H.O. NO.: T 96-44

Standard Method of Test for

ABRASION OF COARSE AGGREGATE BY USE OF THE LOS ANGELES MACHINE[1]

A.S.T.M. Designation: C 131 – 44

ADOPTED, 1939; REVISED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 131; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method covers the procedure for testing crushed rock, crushed slag, uncrushed gravel, and crushed gravel (Note), for resistance to abrasion in the Los Angeles testing machine with an abrasive charge.

NOTE.—Ledge rock, hand-broken into approximately cubical fragments of the different sizes shown, when tested by this method, has been found to have a loss of approximately 85 per cent of that for crushed rock of the same quality

Apparatus

2 (*a*) *Los Angeles Machine.*—The Los Angeles abrasion testing machine, conforming in all its essential characteristics to the design shown in Fig. 1, shall be used. The machine shall consist of a hollow steel cylinder, closed at both ends, having an inside diameter of 28 in. and an inside length of 20 in. The cylinder shall be mounted on stub shafts attached to the ends of the cylinder but not entering it, and shall be mounted in such a manner that it may be rotated about its axis in a horizontal position. An opening in the cylinder shall be provided for the introduction of the test sample. The opening shall be closed dust-tight with a removable cover bolted in place. The cover shall be so designed as to maintain the cylindrical contour of the interior surface unless the shelf is so located that the charge will not fall on the cover, or come in contact with it during the test. A removable steel shelf, projecting radially 3½ in. into the cylinder and extending its full length, shall be mounted along one element of the interior surface of the cylinder. The shelf shall be of such thickness and so mounted, by bolts or other approved means, as to be firm and rigid. The position of the shelf shall be such that the distance from the shelf to the opening, measured along the circumference of the cylinder in the direction of rotation, shall not be less than 50 in.

NOTE.—The use of a shelf of wear-resistant steel, rectangular in cross-section and mounted independently of the cover, is preferred. How-

[1] Under the standardization procedure of the Society, this method is under the joint jurisdiction of the A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates and Committee D-4 on Road and Paving Materials.

[2] Prior to adoption as standard, this method was published as tentative from 1937 to 1939, being revised in 1938 and 1939.

ever, a shelf consisting of a section of rolled angle, properly mounted on the inside of the cover plate, may be used provided the direction of rotation is such that the charge will be caught on the outside face of the angle.

(*b*) *Sieves.*—Square-hole sieves of the following sizes shall conform to the

Abrasive Charge

3. (*a*) The abrasive charge shall consist of cast-iron spheres or steel spheres approximately $1\frac{7}{8}$ in. in diameter and each weighing between 390 and 445 g. The cast-iron spheres shall conform to

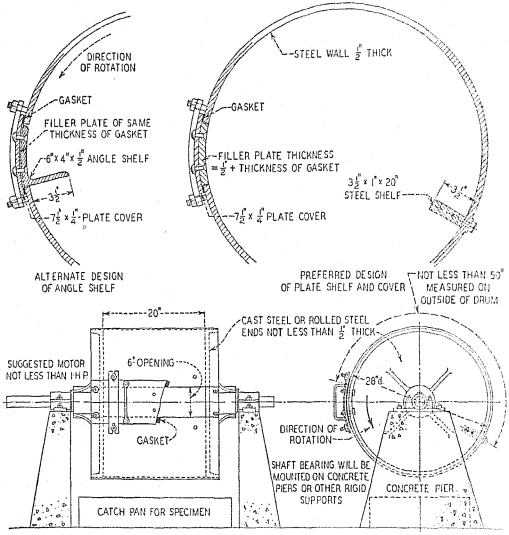


FIG. 1.—Los Angeles Abrasion Testing Machine.

Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials:[3]

Fine Series:
No. 12 (1680-micron)
Coarse Series:
$1\frac{1}{2}$-in.
1-in.
$\frac{3}{4}$-in.
$\frac{1}{2}$-in.
$\frac{3}{8}$-in.

the following requirements as to chemical composition:

Combined carbon, min., per cent	2.50
Graphitic carbon, max., per cent	0.25
Manganese, max., per cent	0.50
Phosphorus, max., per cent	0.25
Sulfur, max., per cent	0.08
Silicon, per cent	1.00

NOTE.—Cast-iron spheres, uniform in size, conforming to these specifications are available. The above chemical requirements for the cast-iron spheres are identical with those for the cast-iron spheres used in the rattler test for paving brick as prescribed in the Standard Specifications for Paving Brick (A.S.T.M. Designa-

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

tion: C 7) of the American Society for Testing Materials.[3]

Steel spheres 1 37/32 in. in diameter and weighing 417 g. can be obtained from at least one manufacturer. If they are not readily available, the next larger size (1 1/8 in.) may be ground to size.

(*b*) The abrasive charge, depending upon the grading of the test sample as described in Section 4, shall be as follows:

Grading	Number of Spheres	Weight of Charge, g.
A	12	5000 ± 25
B	11	4584 ± 25
C	8	3330 ± 20
D	6	2500 ± 15

Test Sample

4. The test sample shall consist of 5000 g. of clean, dry aggregate and shall conform to one of the gradings shown in Table I. The grading used shall be that most nearly representing the aggregate furnished for the work.

TABLE I.—GRADINGS OF TEST SAMPLES.

Sieve Size (Square Openings)		Weight, g.			
Passing	Retained on	Grading A	Grading B	Grading C	Grading D
1½ in.	1 in.	1250	....	....	....
1 in.	¾ in.	1250	....	....	....
¾ in.	½ in.	1250	2500	....	....
½ in.	⅜ in.	1250	2500	....	....
⅜ in.	No. 3	....	....	2500	....
No. 3	No. 4	....	....	2500	....
No. 4	No. 8	....	....	....	5000

Procedure

5. The test sample and the abrasive charge shall be placed in the Los Angeles abrasion testing machine and the machine rotated for 500 revolutions at a speed of from 30 to 33 r.p.m. If an angle is used as the shelf, the machine shall be rotated in such a direction that the charge is caught on the outside surface of the angle. At the completion of the test, the material shall be removed from the machine and sieved on a No. 12 (1680-micron) sieve. The material retained on the sieve shall be washed, dried, and accurately weighed to the nearest gram (Note).

NOTE.—Attention is called to the fact that valuable information concerning the uniformity of the sample under test may be obtained by determining the loss after 100 revolutions. When this determination is made, care should be taken to avoid loss of any part of the sample; the entire sample, including the dust of abrasion, shall be returned to the testing machine for the completion of the test.

Calculation

6. The difference between the original weight and the final weight of the test sample shall be expressed as a percentage of the original weight of the test sample. This value shall be reported as the percentage of wear.

Standard Method of Test for
CLAY LUMPS IN AGGREGATES[1]

A.S.T.M. Designation: C 142 - 39

ADOPTED, 1939.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 142; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test covers the procedure for the approximate determination of clay lumps in the routine examination of aggregates.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Balance.*—Balance or scale sensitive to within 0.1 per cent of the weight of the sample to be weighed.

(*b*) *Containers.*—Containers of a size and shape that will permit the spreading of the sample on the bottom in a thin layer.

(*c*) *Sieves.*—Sieves conforming to the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials.[3]

Samples

3. (*a*) Samples shall be obtained by quartering or by the use of a sampler from a representative sample selected from the material to be tested. They shall be handled in such a manner as to avoid breaking up clay lumps which may be present.

(*b*) Samples shall be dried to substantially constant weight at a temperature not exceeding 110 C. (230 F.).

(*c*) Samples of fine aggregate shall consist of the particles coarser than a No. 16 (1190-micron) sieve and shall weigh not less than 100 g.

(*d*) Samples of coarse aggregate shall be separated into different sizes using the following sieves: No. 4 (4760-micron), $\frac{3}{8}$-in., $\frac{3}{4}$-in., and $1\frac{1}{2}$-in. The weight of sample for the different sizes shall be not less than indicated in the following table:

Size of Particles Making up Sample	Weight of Sample, min., g.
No. 4 to $\frac{3}{8}$-in.	1000
$\frac{3}{8}$ to $\frac{3}{4}$-in.	2000
$\frac{3}{4}$ to $1\frac{1}{2}$-in.	3000
Over $1\frac{1}{2}$-in.	5000

(*e*) In the case of mixtures of fine and coarse aggregates, the material shall be separated into two sizes on the No. 4 (4760-micron) sieve, and the samples of fine and coarse aggregates shall be prepared in accordance with Paragraphs (*c*) and (*d*).

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates.

[2] Prior to adoption as standard, this method was published as tentative from 1938 to 1939.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Procedure

4. The sample shall be spread in a thin layer on the bottom of the container and examined for clay lumps. Any particles which can be broken into finely divided particles with the fingers shall be classified as clay lumps. After all discernible clay lumps have been broken, the residue from the clay lumps shall be removed by use of the sieves indicated in the following table:

Size of Particles Making up Sample	Size of Sieve for Sieving Residue of Clay Lumps
Fine aggregate (retained on No. 16 (1190-micron) sieve)	No. 20 (840-micron)
No. 4 to ⅜-in.	No. 8 (2380-micron)
⅜ to ¾-in.	No. 4 (4760-micron)
¾ to 1½-in.	No. 4 (4760-micron)
Over 1½-in.	No. 4 (4760-micron)

Calculation

5. The percentage of clay lumps shall be calculated to the nearest 0.1 per cent in accordance with the following formula:

$$L = \frac{W - R}{W} \times 100$$

where:

L = percentage of clay lumps,

W = weight of sample, and

R = weight of sample after removal of clay lumps.

Standard Method of Test for
COAL AND LIGNITE IN SAND[1]



A.S.T.M. Designation: C 123 - 44

ADOPTED, 1939; REVISED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 123; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test covers a procedure for the approximate determination of coal and lignite in the routine laboratory examination of sands. This method separates along with the coal and lignite other particles of low specific gravity, such as small pieces of wood, vegetable matter, etc.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Balance.*—A balance having a capacity of at least 200 g. and sensitive to 0.01 g. A less sensitive balance may be used in weighing the wet sample.

(*b*) *Container.*—A container suitable for drying the sand sample.

(*c*) *Beakers.*—Two 400-ml. tall-form, lipped beakers.

(*d*) *Wire Gauze or Sieve.*—Wire gauze or a small sieve having about thirty openings per inch. A piece of wire gauze would probably be more suitable than a sieve.

(*e*) *Hot Plate or Oven.*

Procedure

3. A quantity in excess of 200 g. of sand (sampled wet) shall be dried to constant weight at a temperature of approximately 105 C. A 200-g. sample of the dried sand shall then be weighed to the nearest 0.01 g. The sample shall then be poured slowly into about 250 ml. of a liquid having a specific gravity of 2.0 (Note) contained in one of the 400-ml. beakers. The liquid shall then be poured off into the second beaker passing it through the gauze or sieve. Care shall be taken that only the floating particles are poured off with the liquid and that none of the sand is decanted onto the gauze or sieve. The liquid collected in the second beaker shall then be returned to the beaker containing the sand, and, after further agitation of the sample by stirring, the decanting process just described shall be repeated until the sample is free of floating particles. The decanted par-

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates.

[2] Prior to adoption as standard, this method was published as tentative from 1936 to 1939.

ticles contained on the gauze or sieve shall be washed in carbon tetrachloride, until the flotation liquid is removed, and then dried. The particles will dry very quickly, but may be placed in an oven at 105 C. for a few minutes if desired. The decanted particles shall then be brushed from the gauze or sieve onto the balance pan and the weight accurately determined to the nearest 0.01 g.

NOTE.—Such a liquid can be prepared from an appropriate mixture of carbon tetrachloride and acetylene tetrabromide or bromoform and monobromo-benzene.

Calculation

4. The approximate percentage of coal and lignite shall be calculated from the following formula:

$$\text{Coal and lignite, per cent} = \frac{\text{Wt. of decanted particles}}{\text{Wt. of dry sample (200 g.)}} \times 100$$

Standard Method of Test for

ORGANIC IMPURITIES IN SANDS FOR CONCRETE[1]

A.S.T.M. Designation: C 40 – 33

ADOPTED, 1922; REVISED, 1927, 1933.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 40; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test covers the procedure for an approximate determination of the presence of injurious organic compounds in natural sands which are to be used in cement mortar or concrete. The principal value of the test is to furnish a warning that further tests of the sands are necessary before they are approved for use.

Sample

2. A representative test sample of sand weighing about 1 lb. shall be obtained by quartering or by the use of a sampler.

Reference Standard Color Solution

3. A reference standard color solution shall be prepared by adding 2.5 ml. of a 2 per cent solution of tannic acid in 10 per cent alcohol to 97.5 ml. of a 3 per cent sodium hydroxide[3] solution. This shall be placed in a 12-oz. bottle, stoppered, shaken vigorously, and allowed to stand for 24 hr.

Procedure

4. (*a*) A 12-oz. graduated clear glass bottle shall be filled to the 4½-oz. mark with the sample of the sand to be tested.

(*b*) A 3 per cent solution of sodium hydroxide[3] in water shall be added until the volume of the sand and liquid indicated after shaking is 7 liquid ounces.

(*c*) The bottle shall be stoppered, shaken vigorously, and then allowed to stand for 24 hr.

Determination of Color Value

5. (*a*) After standing 24 hr., the color of the clear liquid above the sample shall be compared with the color of the reference standard color solution prepared at the same time and in accordance with Section 3, or with a glass having a color similar to the color of the reference standard solution. Solutions darker in color than the reference standard color have a "color value" higher than 500 ppm. in terms of tannic acid.

(*b*) In lieu of the method described in Paragraph (*a*), the color of the clear liquid above the sample may be compared with the colors given in Figs. 1 to 5 (the accompanying Color Plate I).

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates.

[2] Prior to adoption as standard, this method was published as tentative from 1921 to 1922. Editorially revised and rearranged in 1939.

[3] Where chemically pure sodium hydroxide is not available, commercial soda lye may be used.

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1944 BOOK OF A.S.T.M. STANDARDS, PART I

STANDARD METHOD OF TEST FOR
ORGANIC IMPURITIES IN SANDS FOR CONCRETE
A.S.T.M. DESIGNATION: C 40

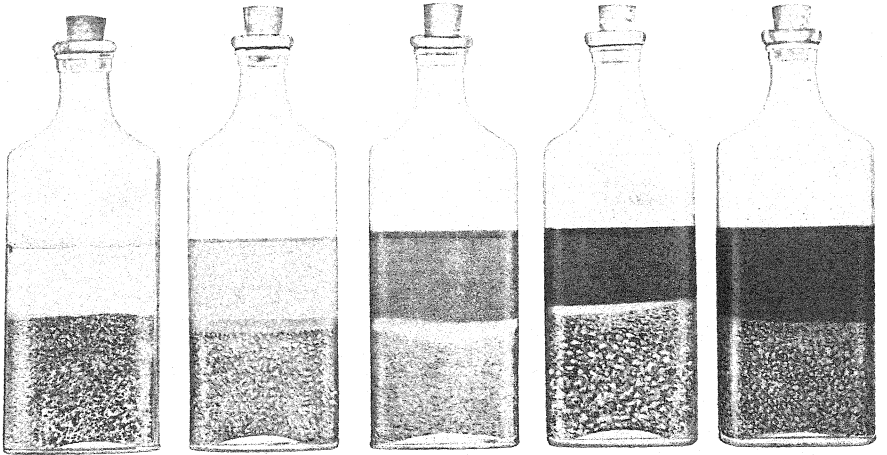


FIG. 1	FIG. 2	FIG. 3	FIG. 4	FIG. 5
Sands suitable for use in high-grade concrete.	Sands which may be used in unimportant concrete work.	Sands which should never be used in concrete.	An unusually bad sand, soil, or loam.	

COLORS OF TREATED SANDS WITH SUGGESTED RANGES OF APPLICATION

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: A37.4-1943
AMERICAN ASSOCIATION STATE
HIGHWAY OFFICIALS STANDARD
A.A.S.H.O. NO.: T 11-42

Standard Method of Test for

AMOUNT OF MATERIAL FINER THAN NO. 200 SIEVE IN AGGREGATES[1]

A.S.T.M. Designation: C 117 - 37

ADOPTED, 1937.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 117; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test outlines the procedure for determining the total quantity of material finer than a standard No. 200 (74-micron) sieve in aggregates.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Sieves.*—A nest of two sieves, the first being a No. 200 (74-micron) sieve conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials,[3] and the second, a sieve having approximately 16 meshes per linear inch.

(*b*) *Container.*—A pan or vessel of a size sufficient to contain the sample covered with water and to permit of vigorous agitation without inadvertent loss of any part of the sample or water.

Test Sample

3. The test sample shall be selected from material which has been thoroughly mixed and which contains sufficient moisture to prevent segregation. A representative sample, sufficient to yield not less than the appropriate weight of dried material, as shown in the following table, shall be selected:

NOMINAL DIAMETER OF LARGEST PARTICLE, IN.	APPROXIMATE MINIMUM WEIGHT OF SAMPLE, KG.
$\frac{1}{4}$	0.5
$\frac{3}{4}$	2.5
$1\frac{1}{2}$ or over	5.0

Procedure

4. (*a*) The test sample shall be dried to constant weight at a temperature not exceeding 110 C. (230 F.) and weighed to the nearest 0.02 per cent.

(*b*) The test sample after being dried and weighed shall be placed in the con-

[1] Under the standardization procedure of the Society, this method is under the joint jurisdiction of A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates and Committee D-4 on Road and Paving Materials.

[2] Prior to adoption as standard, this method was published as tentative from 1935 to 1937.

This method is in effect a revision of and replaces the former Standard Method of Test for Quantity of Clay and Silt in Gravel for Highway Construction (D 72 - 21) and the Standard Method of Decantation Test for Sand and Other Fine Aggregates (D 136 - 28) which methods were accordingly discontinued in 1937.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

tainer and sufficient water added to cover it. The contents of the container shall be agitated vigorously and the wash water poured immediately over the nested sieves, arranged with the coarser sieve on top.

(*c*) The agitation should be sufficiently vigorous to result in the complete separation from the coarse particles of all particles finer than the No. 200 (74-micron) sieve and bring the fine material into suspension in order that it will be removed by decantation of the wash water. Care shall be taken to avoid, as much as possible, the decantation of the coarse particles of the sample. The operation shall be repeated until the wash water is clear.

(*d*) All material retained on the nested sieves shall be returned to the washed sample. The washed aggregate shall be dried to constant weight at a temperature not exceeding 110 C. (230 F.) and weighed to the nearest 0.02 per cent.

Calculation

5. The results shall be calculated from the following formula:

$$\text{Percentage of material finer than No. 200 sieve} = \frac{\text{orig. dry wt.} - \text{dry wt. after washing}}{\text{orig. dry wt.}} \times 100$$

Check Determinations

6. When check determinations are desired, the wash water shall be either evaporated to dryness or filtered through tared filter paper which shall subsequently be dried, the residue weighed, and the percentage calculated from the following formula:

$$\text{Percentage of material finer than No. 200 sieve} = \frac{\text{wt. of residue}}{\text{orig. dry wt.}} \times 100$$

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A.A.S.H.O. NO.: T 27-42

Standard Method of Test for

SIEVE ANALYSIS OF FINE AND COARSE AGGREGATES[1]

A.S.T.M. Designation: C 136 - 39

ADOPTED, 1939.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 136; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test covers a procedure for the determination of the particle size distribution of fine and coarse aggregates, using sieves with square openings. The method is also applicable to the use of laboratory screens with round openings. It is not intended for use in the sieve analysis of aggregates recovered from bituminous mixtures or for the sieve analysis of mineral fillers.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Balance.*—The balance or scale shall be sensitive to within 0.1 per cent of the weight of the sample to be tested.

(*b*) *Sieves.*—The sieves with square openings shall be mounted on substantial frames constructed in a manner that will prevent loss of material during sieving. Suitable sieve sizes shall be selected to furnish the information required by the specifications covering the material to be tested. The woven wire cloth sieves shall conform to the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials.[3]

NOTE.—If round-hole perforated plate screens are used, the openings shall conform to the applicable dimensions and tolerances prescribed in the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials.[3]

Samples

3. (*a*) Samples for sieve analysis shall be obtained, by quartering or by use of a sampler, from a representative sample selected from the material to be tested.

(*b*) Samples of fine aggregate for sieve analysis shall weigh, after drying, not

[1] Under the standardization procedure of the Society, this method is under the joint jurisdiction of A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates and Committee D-4 on Road and Paving Materials.

[2] Prior to adoption as standard, this method was published as tentative from 1938 to 1939.

This method represents a consolidation of the former Standard Methods of Mechanical Analysis of Sand or Other Fine Highway Material, Except Fine Aggregates Used in Cement Concrete (D 7 - 27), of Mechanical Analysis of Broken Stone or Broken Slag, Except Aggregates Used in Cement Concrete (D 18 - 16), of Mechanical Analysis of Mixtures of Sand or Other Fine Material with Broken Stone or Broken Slag, Except Aggregates Used in Cement Concrete (D 19 - 16), and Test for Sieve Analysis of Aggregates for Concrete (C 41 - 36), which standards were accordingly discontinued in 1938.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

less than the amount indicated in the following table:

Material with at least 95 per cent finer than a No. 10 (2000-micron) sieve.... 100 g.
Material with at least 90 per cent finer than a No. 4 (4760-micron) sieve and more than 5 per cent coarser than a No. 10 (2000-micron) sieve.....500 g.

(*c*) Samples of coarse aggregate for sieve analysis shall weigh, after drying, not less than an amount indicated in the following table:

Nominal Maximum Size of Particle, in.	Minimum Weight of Sample, g.[a]
⅜	1 000
½	2 500
¾	5 000
1	10 000
1½	15 000
2	20 000
2½	25 000
3	30 000
3½	35 000

[a] For samples weighing 5000 g. or more it is recommended that sieves mounted in frames 16 in. in diameter or larger be used.

(*d*) In the case of mixtures of fine and coarse aggregates, the material shall be separated into two sizes on the No. 4 (4760-micron) sieve and the samples of fine and coarse aggregates shall be prepared in accordance with Paragraphs (*b*) and (*c*).

(*e*) In the case of fine aggregate, the material finer than the No. 200 (74-micron) sieve shall be determined in accordance with the Standard Method of Test for Amount of Material Finer than No. 200 Sieve in Aggregates (A.S.T.M. Designation: C 117) of the American Society for Testing Materials[3] and the sieve analysis made on the material coarser than the No. 200 (74-micron) sieve.

Preparation of Sample

4. Samples shall be dried to substantially constant weight at a temperature not exceeding 110 C. (230 F.).

Procedure

5. (*a*) The sample shall be separated into a series of sizes using such sieves as are necessary to determine compliance with the specifications for the material under test. The sieving operation shall be conducted by means of a lateral and vertical motion of the sieve, accompanied by jarring action so as to keep the sample moving continuously over the surface of the sieve. In no case shall fragments in the sample be turned or manipulated through the sieve by hand. Sieving shall be continued until not more than 1 per cent by weight of the residue passes any sieve during 1 min. On that portion of the sample retained on the No. 4 (4760-micron) sieve, the above described procedure for determining thoroughness of sieving shall be carried out with a single layer of material. When mechanical sieving is used, the thoroughness of sieving shall be tested by using the hand method of sieving as described above.

(*b*) The weight of each size shall be determined on a scale or balance conforming to the requirements specified in Section 2 (*a*).

Report

6. The results of the sieve analysis shall be reported as follows: (*a*) total percentages passing each sieve, or (*b*) total percentages retained on each sieve, or (*c*) percentages retained between consecutive sieves, depending upon the form of the specifications for the use of the material under test. Percentages shall be reported to the nearest whole number and shall be calculated on the basis of the weight of the test sample including any material finer than the No. 200 (74-micron) sieve.

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A.A.S.H.O. NO.: T 85-42

Standard Method of Test for

SPECIFIC GRAVITY AND ABSORPTION OF COARSE AGGREGATE[1]

A.S.T.M. Designation: C 127 - 42

ADOPTED, 1939; REVISED, 1942.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 127; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. (*a*) This method of test is intended for use in making determinations of bulk and apparent specific gravity, and absorption (after 24 hr. in water at room temperature) of coarse aggregate. The bulk specific gravity is the value generally desired for calculations in connection with portland-cement concrete.

(*b*) This method determines directly the bulk specific gravity as defined in the Standard Definitions of Terms Relating to Specific Gravity (A.S.T.M. Designation: E 12) of the American Society for Testing Materials,[3] or the bulk specific gravity on the basis of weight of saturated surface-dry aggregate, or the apparent specific gravity as defined in the Standard Definitions E 12.[3]

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Balance.*—A balance having a capacity of 5 kg. or more and sensitive to 0.5 g. or less.

(*b*) *Wire Basket.*—A wire basket of No. 4 mesh, approximately 8 in. in diameter and 8 in. in height.

(*c*) A suitable container for immersing the wire basket in water and suitable apparatus for suspending the wire basket from center of scale pan of balance.

Sample

3. Approximately 5 kg. of the aggregate shall be selected from the sample to be tested by the method of quartering, rejecting all material passing a $\frac{3}{8}$-in. sieve. In the case of homogeneous aggregate, all material may be retained on a 1-in. sieve.

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[1] Under the standardization procedure of the Society, this method is under the joint jurisdiction of the A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates and Committee D-4 on Road and Paving Materials.

[2] Prior to adoption as standard, this method was published as tentative from 1936 to 1939.

This method represents a consolidation of the former Standard Methods of Test for Apparent Specific Gravity of Coarse Aggregates (D 30 - 18), Test for Absorption by Aggregates for Concrete (Laboratory Determination) (C 93 - 36), Field Test for Absorption of Mixing Water by Aggregates for Concrete (C 96 - 36), and the former Tentative Method of Test for Apparent Specific Gravity of Coarse Aggregates in a Saturated Condition (C 86 - 31 T), which methods were accordingly discontinued in 1936.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Procedure

4. (*a*) After thoroughly washing to remove dust or other coatings from the surface of the particles, the sample shall be dried to constant weight at a temperature of 100 to 110 C. (Note) and then immersed in water at 15 to 25 C., for a period of 24 hr. It shall then be removed from the water and rolled in a large absorbent cloth until all visible films of water are removed, although the surfaces of the particles still appear to be damp. The larger fragments may be individually wiped. Care should be taken to avoid evaporation during the operation of surface drying. The weight of the sample in the saturated surface-dry condition shall then be obtained. This and all subsequent weights shall be determined to the nearest 0.5 g.

NOTE.—Where the absorption and specific gravity values may be utilized as a basis for designing concrete mixtures with aggregates normally used in a moist condition, the requirement of drying to constant weight may be eliminated.

(*b*) After weighing, the saturated surface-dry sample shall be placed immediately in the wire basket and its weight in water determined.

(*c*) The sample shall then be dried to constant weight at a temperature of 100 to 110 C., cooled to room temperature, and weighed.

Bulk Specific Gravity

5. The bulk specific gravity as defined in the Standard Definitions of Terms Relating to Specific Gravity (A.S.T.M. Designation: E 12) of the American Society for Testing Materials,[3] shall be calculated from the following formula:

$$\text{Bulk sp. gr.} = \frac{A}{B - C}$$

where:

A = weight in grams of oven-dry sample in air,
B = weight in grams of saturated surface-dry sample in air, and
C = weight in grams of saturated sample in water.

Bulk Specific Gravity (Saturated Surface-Dry Basis)

6. The bulk specific gravity on the basis of weight of saturated surface-dry aggregate shall be calculated from the following formula:

$$\text{Bulk sp. gr. (saturated surface-dry basis)} = \frac{B}{B - C}$$

Apparent Specific Gravity

7. The apparent specific gravity as defined in the Standard Definitions of Terms Relating to Specific Gravity (A.S.T.M. Designation: E 12) of the American Society for Testing Materials,[3] shall be calculated from the following formula:

$$\text{Apparent sp. gr.} = \frac{A}{A - C}$$

Absorption

8. The percentage of absorption shall be calculated from the following formula:

$$\text{Absorption, per cent} = \frac{B - A}{A} \times 100$$

Reproducibility of Results

9. Duplicate determinations should check to within 0.02 in the case of specific gravity and 0.05 per cent in the case of percentage of absorption.

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Standard Method of Test for

SPECIFIC GRAVITY AND ABSORPTION OF FINE AGGREGATE[1]

A.S.T.M. Designation: C 128 - 42

ADOPTED, 1939; REVISED, 1942.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 128; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. (*a*) This method of test is intended for use in making determinations of bulk and apparent specific gravity, and absorption (after 24 hr. in water at room temperature) of fine aggregate. The bulk specific gravity is the value generally desired for calculations in connection with portland-cement concrete.

(*b*) This method determines directly the bulk specific gravity as defined in the Standard Definitions of Terms Relating to Specific Gravity (A.S.T.M. Designation: E 12) of the American Society for Testing Materials,[3] or the bulk specific gravity on the basis of weight of saturated surface-dry aggregate, or the apparent specific gravity as defined in the Standard Definitions E 12.[3]

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Balance.*—A balance having a capacity of 1 kg. or more and sensitive to 0.1 g. or less.

(*b*) *Flask.*—A volumetric flask of 500-ml. capacity, calibrated to 0.15 ml. at 20 C.

(*c*) *Conical Mold.*—A conical metal mold $1\frac{1}{2}$ in. in diameter at the top, $3\frac{1}{2}$ in. in diameter at the bottom, and $2\frac{3}{4}$ in. in height.

(*d*) *Tamping Rod.*—A metal tamping rod weighing 12 oz. and having a flat circular tamping face 1 in. in diameter.

Preparation of Sample

3. Approximately 1000 g. of the fine aggregate selected from the sample by the method of quartering shall be placed, in a suitable pan or vessel, after drying to constant weight at a temperature of 100 to 110 C. (Note 1), covered with water, and permitted to stand for 24 hr. The sample shall then be spread on a flat surface, exposed to a gently moving current of warm air, and stirred frequently to secure uniform drying. This operation shall be continued until the fine aggregate approaches a free-flowing condition. The fine aggregate shall then be

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[1] Under the standardization procedure of the Society, this method is under the joint jurisdiction of the A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates and Committee D-4 on Road and Paving Materials.

[2] Prior to adoption as standard, this method was published as tentative from 1936 to 1939.

This method represents a consolidation of the former Standard Methods of Test for Approximate Apparent Specific Gravity of Fine Aggregate (C 65 - 30), Test for Apparent Specific Gravity of Sand, Stone, and Slag Screenings, and other Fine Non-Bituminous Highway Materials (D 55 - 25), Test for Absorption by Aggregates for Concrete (Laboratory Determinations) (C 95 - 36), and Field Test for Absorption of Mixing Water by Aggregates for Concrete (C 96 - 36), which methods were accordingly discontinued.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

placed loosely in the conical mold, the surface lightly tamped 25 times with the metal rod, and the mold lifted vertically. If free moisture is present, the cone of fine aggregate will retain its shape. Drying with constant stirring shall be continued and tests made at frequent intervals, until the cone of fine aggregate slumps upon removal of the mold. This indicates that the fine aggregate has reached a surface-dry condition (Note 2).

NOTE 1.—Where the absorption and specific gravity values may be utilized as a basis for designing concrete mixtures with aggregates normally used in a moist condition, the requirement of drying to constant weight may be eliminated.

NOTE 2.—The procedure described in Section 3 is intended to insure that the first trial determination shall be made with some free water in the sample. If the cone of fine aggregate slumps on the first trial, the fine aggregate has been dried past the saturated and surface-dry condition. In this case a few milliliters of water shall be thoroughly mixed with the fine aggregate and the sample permitted to stand in a covered container for 30 min. The process of drying and testing the fine aggregate shall then be resumed.

Procedure

4. (*a*) A 500.0-g. sample of the material, prepared as described in Section 3, shall be introduced immediately into the flask and the flask filled almost to the 500-ml. mark with water at a temperature of 20 C. The flask shall then be rolled on a flat surface to eliminate all air bubbles, after which it shall be placed in a constant temperature bath maintained at 20 C. After approximately 1 hr. it shall be filled with water to the 500-ml. mark and the total weight of water (Note) introduced into the flask shall be determined to the nearest 0.1 g.

NOTE.—If desired, the quantity of water necessary to fill the flask may be determined volumetrically by the use of a burette accurate to 0.1 ml.

(*b*) The fine aggregate shall be removed from the flask and dried to constant weight at a temperature of 100 to 110 C., cooled to room temperature in a desiccator, and weighed.

Bulk Specific Gravity

5. The bulk specific gravity as defined in the Standard Definitions of Terms Relating to Specific Gravity (A.S.T.M. Designation: E 12) of the American Society for Testing Materials,[3] shall be calculated from the following formula:

$$\text{Bulk sp. gr.} = \frac{A}{V - W}$$

where:

A = weight in grams of oven-dry sample in air,
V = volume in milliliters of flask, and
W = weight in grams or volume in milliliters of water added to flask.

Bulk Specific Gravity (Saturated Surface-Dry Basis)

6. The bulk specific gravity on the basis of weight of saturated surface-dry aggregate shall be calculated from the following formula:

$$\text{Bulk sp. gr. (saturated surface-dry basis)} = \frac{500}{V - W}$$

Apparent Specific Gravity

7. The apparent specific gravity as defined in the Standard Definitions of Terms Relating to Specific Gravity (A.S.T.M. Designation: E 12) of the American Society for Testing Materials,[3] shall be calculated from the following formula:

$$\text{Apparent sp. gr.} = \frac{A}{(V - W) - (500 - A)}$$

Absorption

8. The percentage of absorption shall be calculated from the following formula:

$$\text{Absorption, per cent} = \frac{500 - A}{A} \times 100$$

Reproducibility of Results

9. Duplicate determinations should check to within 0.02 in the case of specific gravity and 0.05 per cent in the case of percentage of absorption.

Standard Method of Test for

STRUCTURAL STRENGTH OF FINE AGGREGATE USING CONSTANT WATER-CEMENT-RATIO MORTAR[1]

A.S.T.M. Designation: C 87 - 44

ADOPTED, 1936; REVISED, 1939, 1942, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 87; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test covers the procedure for determining the "structural strength" of sands to be used as fine aggregate in concrete by means of a compression test on a mortar of plastic consistency and gaged to a definite water-cement ratio.

Mortar

2. Cement and water (Section 5) in quantities[3] that will give a water-cement ratio of 0.6 by weight shall be placed in an appropriate vessel[4] and the cement permitted to absorb water for 1 min. The materials shall then be mixed with a spoon[4] into a smooth paste. A sample of sand of known weight[5] that has been placed in a saturated surface-dry condition[5] by means of the procedure specified in Section 3 of the Standard Method of Test for Specific Gravity and Absorption of Fine Aggregate (A.S.T.M. Designation: C 128) of the American Society for Testing Materials,[6] shall be beaten into the mixture until the material appears to be of the desired consistency (flow 100 ± 5). The mixing shall be continued for 30 sec. and a determination of the flow made in accordance with Section 3.

Flow Test

3. (*a*) *Apparatus.*—The flow test shall be made on a 10-in. flow table consisting of a circular bronze plate 10 in. in diameter, so mounted that the surface is level, and equipped with a mechanism for raising the plate ½ in. and allowing it to fall without cushioning. The flow table with the attached shaft shall weigh 9 ± 1 lb. The frame of the table shall

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates.

[2] Prior to adoption as standard, this method was published as tentative from 1931 to 1936, being revised in 1932.

[3] For six 2-in. cubes, 600 g. of cement and 360 ml. of water will usually give sufficient mortar. The quantity of sand used with this amount of cement may vary from 1200 g. for fine sand to 2000 g. or more for coarse sand.

[4] An ordinary sauce pan of 4-qt. capacity, 9½ in. in diameter at the top and 4½ in. in height, and a large iron kitchen spoon are suitable for use in mixing the mortar.

[5] If the absorption as determined by the Standard Method C 128 is known, sand may be prepared for test by adding to a known weight of dry sand the amount of water it will absorb, mixing thoroughly, and permitting the sand to stand in a covered pan for 30 min. before use.

[6] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

be attached rigidly to a concrete pedestal which in turn shall be attached rigidly to the floor. The concrete pedestal shall be at least 8 in. in diameter and 25 in. in height and shall weigh at least 100 lb. A right truncated cone, $2\frac{3}{4}$ in. in diameter at the top, 4 in. in diameter at the bottom, and 2 in. in height shall be used to measure the volume of the mortar for the test.

(*b*) *Procedure.*—Immediately after mixing, the mortar shall be placed in the cone which has been carefully centered on the flow table. The mortar shall be rodded 25 times with a $\frac{3}{8}$-in. rod, the surplus struck off, the plate wiped clean, the cone removed, and the table given 10 drops in 10 sec. The percentage of increase in the diameter of the base of the pat, measured on two diameters at right angles to each other, shall be recorded as the flow. Should the flow be too great, the mortar may be returned to the mixing vessel, additional sand added, and another determination of the flow made. If more than two trials must be made to obtain a flow of 100 ± 5, the mortar should be considered as a trial mortar and the test specimens prepared from a new batch. If the mortar is too dry, the batch shall be discarded. The quantity of sand used shall be determined by subtracting the weight of the portion remaining after mixing from the weight of the initial sample.

Molding Specimens

4. Immediately following the determination of the flow, the mortar shall be placed in 2 by 4-in. cylindrical molds in three layers or in 2-in. cubes in two layers, each layer rodded in place with 25 strokes of a $\frac{3}{8}$-in. rod, and the mold filled to overflowing. The specimens shall be placed in a moist closet for curing. Three to four hours after molding the specimens shall be struck off to a smooth surface. Twenty to twenty-four hours after molding, the specimens shall be removed from the molds and stored in water until tested.

Temperature

5. The temperature of the mixing water, moist closet, and storage tank shall be maintained between 65 and 75 F.

Capping Specimens

6. If the specimens are cylindrical, they shall be capped before testing in such a manner that the ends are perfectly plane and at right angles to the axis of the cylinder. The material used for capping and the thickness of the cap shall be such that it will not flow or fracture under the load. Cubes made in suitable molds need not be capped and shall be tested at right angles to the direction of molding.

NOTE.—It is desirable that the capping material have a value for modulus of elasticity equal to or greater than that of the mortar.

Standard Method of Test for
UNIT WEIGHT OF AGGREGATE[1]

A.S.T.M. Designation: C 29 - 42

ADOPTED, 1921; REVISED, 1927, 1939, 1942.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 29; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test covers the procedures for determining the unit weight of fine, coarse, or mixed aggregates.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Balance.*—A balance or scale sensitive to 0.5 per cent of the weight of the sample to be weighed.

(*b*) *Tamping Rod.*—A straight metal rod, $\frac{5}{8}$ in. in diameter and 24 in. in length, with one end tapered for a distance of 1 in. to a blunt bullet-shaped point.

(*c*) *Measure.*—A metal measure, cylindrical in form and preferably provided with handles. It shall be watertight, with the top and bottom true and even, preferably machined to accurate dimensions on the inside, and of sufficient rigidity to retain its form under rough usage. The $\frac{1}{2}$ and 1-cu. ft. measures shall be reinforced around the top with a No. 10 to No. 12 gage steel band $1\frac{1}{2}$ in. in width. The measures required, depending upon the maximum size of the coarsest particles in the aggregate to be tested, shall have capacities of $\frac{1}{10}$, $\frac{1}{2}$, or 1 cu. ft. and shall conform to the following dimensional requirements:

Capacity, cu. ft.	Inside Diameter, in.	Inside Height, in.	Thickness of Metal, U. S. Gage	Size of Largest Particles of Aggregate, in.
$\frac{1}{10}$	6.00	6.10	No. 10 to No. 12	$\frac{1}{2}$
$\frac{1}{2}$	10.00	11.00	No. 10 to No. 12	$1\frac{1}{2}$
1	14.00	11.23	No. 10 to No. 12	4

Calibration of Measure

3. The measure shall be calibrated by accurately determining the weight of water at 16.7 C. (62 F.) required to fill it. The factor for any unit shall be obtained by dividing the unit weight of water at 16.7 C. (62 F.) (62.355 lb. per cu. ft.) by the weight of water at 16.7 C. (62 F.) required to fill the measure.

Sample

4. The sample of aggregate shall be room dry and thoroughly mixed.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates.

[2] Prior to adoption as standard, this method was published as tentative from 1920 to 1921.

Compact Weight Determination

Rodding Procedure

5. The rodding procedure is applicable to aggregates having a maximum size of 2 in. or less.

(*a*) The measure shall be filled one-third full and the top leveled off with the fingers. The mass shall be rodded with the tamping rod with 25 strokes, evenly distributed over the surface. The measure shall be filled two-thirds full and again rodded with 25 strokes as before. The measure shall then be filled to overflowing, rodded 25 times, and the surplus aggregate struck off, using the tamping rod as a straight edge.

(*b*) In rodding the first layer, the rod shall not be permitted to forcibly strike the bottom of the measure. In rodding the second and final layers, only enough force shall be used to cause the tamping rod to penetrate the last layer of aggregate placed in the measure.

(*c*) The net weight of the aggregate in the measure shall be determined. The unit weight of the aggregate shall then be obtained by multiplying the net weight of the aggregate by the factor found as described in Section 3.

Jigging Procedure

6. The jigging procedure is applicable to aggregates having a maximum size greater than 2 in. and not to exceed 4 in.

(*a*) The measure shall be filled in three approximately equal layers as described in Section 5 (*a*), each layer being compacted by placing the measure on a firm foundation, such as a cement-concrete floor, and raising alternate sides of the measure about 2 in. from the foundation and allowing it to drop in such a manner as to hit with a sharp, slapping blow. The aggregate particles, by this procedure, will arrange themselves in a closely compacted condition. Each layer shall be compacted by dropping the measure 50 times in the manner described, 25 times on each side. The surface of the aggregate shall then be leveled off with the fingers or a straightedge in such a way that any slight projections of the larger pieces of the coarse aggregate shall balance the larger voids in the surface below the top of the measure.

(*b*) The net weight of the aggregate in the measure shall be determined. The unit weight of the aggregate shall then be obtained by multiplying the net weight of the aggregate by the factor found as described in Section 3.

Loose Weight Determination

Shoveling Procedure

7. (*a*) The shoveling procedure is applicable to aggregates having a maximum size of 4 in. or less. The measure shall be filled to overflowing by means of a shovel or scoop, the aggregate being discharged from a height of not to exceed 2 in. above the top of the measure. Care shall be taken to prevent, so far as possible, segregation of the particle sizes of which the sample is composed. The surface of the aggregate shall then be leveled off with the fingers or a straightedge in such a way that any slight projections of the larger pieces of the coarse aggregate shall balance the larger voids in the surface below the top of the measure.

(*b*) The net weight of the aggregate in the measure shall be determined. The unit weight of the aggregate shall then be obtained by multiplying the net weight of the aggregate by the factor found as described in Section 3.

Reproducibility of Results

8. Results with the same sample should check within 1 per cent.

APPROVED AS
AMERICAN STANDARD
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ASA NO.: A19-1937

AMERICAN ASSOCIATION STATE
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A.A.S.H.O. NO.: T 20-42

Standard Method of Test for

VOIDS IN AGGREGATE FOR CONCRETE[1]

A.S.T.M. Designation: C 30 - 37

ADOPTED, 1922; REVISED, 1937.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 30; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test outlines the procedure for determining voids in aggregate.

Percentage of Voids

2. The voids in aggregate shall be determined by the following formula:

$$\text{Percentage of voids} = \frac{(\text{sp. gr.} \times 62.355) - \text{Wt.}}{\text{sp. gr.} \times 62.355} \times 100$$

where:

sp. gr. = the bulk specific gravity of the aggregate as determined by the appropriate method referred to in Section 3 (*a*) or (*b*),

62.355 = the weight in pounds of 1 cu. ft. of water at the standard temperature of 16.7 C. (62 F.), and

Wt. = the weight in pounds per cubic foot of the aggregate as determined by the method referred to in Section 3 (*c*).

NOTE.—For complete accuracy, "Wt." should be the weight of the air-dried sample minus the weight of any contained moisture.

Methods of Testing

3. The specific gravity and weight of the aggregate shall be determined in accordance with the following methods of test of the American Society for Testing Materials:

(*a*) *Specific Gravity of Fine Aggregate.*—Standard Method of Test for Specific Gravity and Absorption of Fine Aggregate (A.S.T.M. Designation: C 128),[3] Section 5.

(*b*) *Specific Gravity of Coarse Aggregate.*—Standard Method of Test for Specific Gravity and Absorption of Coarse Aggregate (A.S.T.M. Designation: C 127),[3] Section 5.

(*c*) *Unit Weight of Aggregate.*—Standard Method of Test for Unit Weight of Aggregate (A.S.T.M. Designation: C 29).[3]

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates.

[2] Prior to adoption as standard, this method was published as tentative from 1920 to 1922.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Definition of

THE TERM SAND[1]

A.S.T.M. Designation: C 58 – 28

ADOPTED, 1928.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 58; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Sand.—The fine granular material (usually less than $\frac{1}{4}$ in. in diameter) resulting from the natural disintegration of rock, or from the crushing of friable sandstone rocks.

NOTE.—When used without a qualifying adjective, the term sand is generally understood to mean the product of the natural disintegration of siliceous or calcareous rock. Sand should be distinguished from screenings, gravel, etc. The size of particle and other physical characteristics should be taken care of in specifications. The fine material resulting from the crushing of blast-furnace slag is known as "slag sand."

[1] Under the standardization procedure of the Society, this definition is under the jurisdiction of the A.S.T.M. Committee E-8 on Nomenclature and Definitions.

[2] Prior to adoption as standard, this definition was published as tentative from 1924 to 1928, being revised in 1925 and 1926.

A "tentative revision" of this standard appears in the Tenta-

Standard Specifications for

READY-MIXED CONCRETE[1]

A.S.T.M. Designation: C 94 - 44

ADOPTED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 94; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. (*a*) These specifications cover ready-mixed concrete for general use. Requirements for quality of materials and for proportions and quality of concrete shall be either as herein specified or as specified by the purchaser by reference to applicable general specifications for concrete. Applicable general specifications to which reference is made by the purchaser shall be considered a part of these specifications. In any case where the requirements of such general specifications are in conflict with these specifications the requirements of the general specifications shall govern unless otherwise specified by the purchaser.

(*b*) For the purpose of these specifications, ready-mixed concrete is portland-cement concrete manufactured for delivery to a purchaser in a plastic and unhardened state and delivered in a truck mixer or a truck agitator.

Basis of Purchase

2. (*a*) The basis of purchase shall be the cubic yard of plastic and unhardened concrete as delivered to the purchaser.

(*b*) The volume of plastic and unhardened concrete in a given batch shall be determined in accordance with the Standard Method of Test for Weight per Cubic Foot, Yield, and Air Content (Gravimetric) of Concrete (A.S.T.M. Designation: C 138)[3] based on the total weight of the batch, determined either from the weights of all materials, including water, entering the batch or directly from the net weight of the concrete in the batch as delivered.

Materials

3. (*a*) In the absence of designated applicable specifications covering requirements for quality of materials, the following specifications shall govern:

(*1*) *Portland Cement.*—Portland cement shall conform to the Standard Specifications for Portland Cement (A.S.T.M. Designation: C 150).[3] The purchaser should specify the type required, but if no type is specified, the

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates.

[2] Prior to their present adoption as standard, these specifications were published as tentative from 1933 to 1935. They were adopted in 1935, published as standard from 1935 to 1942, being revised in 1938, but further revised and issued as tentative in 1941, revised in 1942, 1943, and 1944.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

requirements of type I as prescribed in Specifications C 150 shall apply.

(2) *Aggregates.*—Aggregates shall conform to the Standard Specifications for Concrete Aggregates (A.S.T.M. Designation: C 33).[3]

(3) *Water.*—Water shall be clear and free from injurious amounts of oil, acid, alkali, organic matter, or other deleterious substances.

(*b*) Admixtures, if to be used, shall be provided for in the contract or in the designated applicable specifications for concrete.

Quality of Concrete

4. In the absence of designated applicable general specifications, the purchaser shall select one of the two following alternate bases for specifying the quality of the concrete:

Alternate No. 1:

(*a*) When the purchaser requires the manufacturer to assume responsibility for the design of the concrete mixture, the purchaser shall specify the following:

(1) Minimum allowable compressive or flexural strength as determined on samples taken at point of delivery. The basis shall be either the compressive strength at 28 days or the flexural strength at 14 days (Note). The purchaser shall specify the requirements for strength in terms of tests of standard specimens cured under standard laboratory conditions for moist curing. The purchaser, in selecting the minimum allowable compressive or flexural strength to be specified, shall give consideration to requirements for durability, surface texture, and density, in addition to those for structural design.

(2) Designated size, or sizes, of coarse aggregate.

(3) Maximum and minimum allowable slump at point of delivery.

NOTE.—Table I suggests a basis for the selection of minimum allowable compressive strengths which, for average normal portland cement, will result in the use of water-cement ratios that will produce concrete suitable for various types of structures and conditions of exposure. A corresponding table of flexural strengths is not given because of the wider range in values which may be obtained with a given water-cement ratio for different materials.

(*b*) Prior to the execution of the contract, the manufacturer shall furnish a statement to the purchaser, giving the proportions by weight (dry) of cement, fine and coarse aggregates which will be used in the manufacture of each class of concrete ordered by the purchaser. If required, he shall also furnish evidence satisfactory to the purchaser that the proportions selected will produce concrete of the quality specified.

Alternate No. 2:

(*a*) When the purchaser assumes responsibility for the design of the concrete mixture, he shall specify the following:

(1) Cement content in bags per cubic yard of concrete, or equivalent units.

(2) Designated size, or sizes, of coarse aggregate.

(3) Maximum allowable water content in gallons per bag of cement, or equivalent units, including surface moisture, but excluding water of absorption, of the aggregates.

(4) Maximum and minimum allowable slump at point of delivery.

(*b*) Prior to the actual delivery of the concrete, the manufacturer shall furnish a statement to the purchaser giving the proportions by weight (dry) of cement, fine and coarse aggregates which will be used in the manufacture of each class of concrete ordered by the purchaser. Such proportions shall be subject to the approval of the purchaser.

Range in Slump

5. When the maximum allowable slump is 3 in. or less, the difference be-

tween the specified maximum and minimum slumps shall be 1 in. When the maximum allowable slump is greater than 3 in., the difference between the specified maximum and minimum slumps shall be 2 in.

other materials. When cement is measured in bags, no fraction of a bag shall be used unless weighed.

(*b*) Aggregate shall be measured by weight. Batch weights shall be based on dry materials and shall be the required

TABLE I.—COMPRESSIVE STRENGTHS OF CONCRETE SUITABLE FOR VARIOUS CONDITIONS OF EXPOSURE.

NOTE.—Presented as information only and not as a part of the specifications; adapted from Tables 1 and 2 of the Report of the Joint Committee on Standard Specifications for Concrete and Reinforced Concrete, June, 1940.

Type or Location of Structure	Compressive Strength, min., psi. at 28 days							
	Severe or Moderate Climate, Wide Range of Temperatures, Rain and Long Freezing Spells or Frequent Freezing and Thawing				Mild Climate, Rain or Semi-Arid, Rarely Snow or Frost			
	Thin Sections		Moderate Sections		Thin Sections		Moderate Sections	
	Reinforced	Plain	Reinforced	Plain	Heavy and Mass Sections	Reinforced	Plain	Reinforced	Plain	Heavy and Mass Sections
A. At the waterline in hydraulic or waterfront structures or portions of such structures where complete saturation or intermittent saturation is possible, but not where the structure is continuously submerged:										
In sea water	5000	4500			4000	5000	4500			4000
In fresh water	4500	4000			3600	4500	4000			3600
B. Portions of hydraulic or waterfront structures some distance from the waterline, but subject to frequent wetting:										
By sea water	4500	4000			4000	4500	3600			3200
By fresh water	4000	3600			3600	4000	3200			2800
C. Ordinary exposed structures, buildings and portions of bridges not coming under above groups	4000	3600			3200	4000	3200			2800
D. Complete continuous submergence:										
In sea water	4000	3600			3200	4000	3600			3200
In fresh water	3600	3200			2800	3600	3200			2800
E. Concrete deposited through water	....	....	4500		4500	...	...	4500		4500
F. Pavement slabs directly on ground:										
Wearing slabs	4500	4000	...		...	4000	3600	...		...
Base slabs	3600	3200	...		...	3200	2800	...		...

G. Special cases:
(*a*) For concrete exposed to strong sulfate ground waters, or other corrosive liquids or salts, the minimum compressive strength should be 5000 psi.
(*b*) For concrete not exposed to the weather, such as the interior of buildings and portions of structures entirely below ground, no exposure hazard is involved and the compressive strength should be selected on the basis of the structural design requirements.

Measuring Materials

6. (*a*) Cement shall be measured by weight or, if permitted by the purchaser, in bags of 94 lb. each. When cement is measured by weight, it shall be weighed on a scale separate from those used for

weights of dry materials plus the total weight of moisture (both absorbed and surface) contained in the aggregate.

(*c*) Water shall be measured by volume or by weight. The device for the measurement of the water shall be readily adjustable and, under all operating con-

ditions, shall have an accuracy of 1 per cent of the quantity of water required for the batch. The device shall be so arranged that the measurements will not be affected by variable pressures in the water supply line. Measuring tanks shall be equipped with outside taps and valves to provide for checking their calibration unless other means are provided for readily and accurately determining the amount of water in the tank. In the case of truck mixers, if wash water is permitted to be used as a portion of the mixing water for succeeding batches, it shall be accurately measured in a separate tank, provided for the purpose, and taken into account in determining the amount of additional mixing water required.

(*d*) Powdered admixtures shall be measured by weight, and paste or liquid admixtures by weight or volume.

Batching Plant

7. (*a*) Unless otherwise permitted by the purchaser, bins with adequate separate compartments for fine aggregates and for each required size of coarse aggregate shall be provided in the batching plant. Each compartment shall be designed to discharge efficiently and freely into the weighing hopper. Means of control shall be provided so that, as the quantity desired in the weighing hopper is being approached, the material may be added slowly and shut off with precision. A port or other opening for removing an overload of any one of the several materials from the hopper shall be provided. Hoppers shall be constructed so as to eliminate accumulations of tare materials and to discharge fully.

(*b*) The scales for weighing aggregates and cement may be either of the horizontal-beam or of the springless-dial type. They shall be accurate within 1 per cent under operating conditions. Adequate standard test weights shall be available for checking accuracy. All exposed fulcrums, clevises, and similar working parts of scales shall be kept clean. When beam-type scales are used, provision shall be made for indicating to the operator that the required load in the weighing hopper is being approached; the device shall indicate at least the last 200 lb. of load. All weighing and indicating devices shall be in full view of the operator while charging the hopper and he shall have convenient access to all controls.

Mixers and Agitators

8. (*a*) Mixers may be stationary mixers or truck mixers. Agitators may be truck mixers or truck agitators. Each mixer and agitator shall have attached thereto in a prominent place a metal plate on which is plainly marked, for the various uses for which the equipment is designed, the capacity of the drum or container in terms of volume of mixed concrete and the speed of rotation of the mixing drum or blades.

(*b*) The mixer when loaded to capacity shall be capable of combining the ingredients of the concrete within the specified time into a thoroughly mixed and uniform mass and of discharging the concrete with a satisfactory degree of uniformity as indicated in Paragraph (*d*).

(*c*) The agitator when loaded to capacity shall be capable of maintaining the mixed concrete in a thoroughly mixed and uniform mass and of discharging the concrete with a satisfactory degree of uniformity as indicated in Paragraph (*d*).

(*d*) The purchaser may, from time to time, make slump tests of individual samples taken at approximately the one-quarter and the three-quarter points of the load and if the slumps differ by more than 2 in. the mixer or agitator shall not be used unless the condition is corrected or except as provided in Paragraph (*e*).

(*e*) If the requirements of Paragraph (*d*) are not met by mixers operated at the minimum specified mixing time and loaded to capacity, or by agitators when loaded to capacity, the equipment may still be used when operation with a longer mixing time or with a smaller load will permit the requirements of Paragraph (*d*) to be met.

(*f*) Mixers and agitators shall be examined daily for changes in condition due to accumulations of hardened concrete or mortar or to wear of blades. When any such change of condition is found the tests described in Paragraph (*d*) should be repeated.

Mixing and Delivery

9. (*a*) Ready-mixed concrete shall be mixed and delivered to the point designated by the purchaser by means of one of the following combinations of operations:

(*1*) Mixed completely in a stationary mixer and the mixed concrete transported to the point of delivery in a truck agitator or in a truck mixer operating at agitator speed. (Known as *central-mixed concrete.*)

(*2*) Mixed partially in a stationary mixer, and the mixing completed in a truck mixer. (Known as *shrink-mixed concrete.*)

(*3*) Mixed completely in a truck mixer. (Known as *transit-mixed concrete.*)

(*b*) Mixers and agitators shall be operated within the limits of capacity and speed of rotation designated by the manufacturer of the equipment.

(*c*) When a stationary mixer is used for the complete mixing of the concrete, the mixing time for mixers of 1 cu. yd. or less shall not be less than 1 min. For mixers of larger capacities, this minimum shall be increased 15 sec. for each cubic yard or fraction thereof of additional capacity. Mixing time shall be measured from the time all cement and aggregates are in the drum. The batch shall be so charged into the mixer that some water will enter in advance of cement and aggregate, and all water shall be in the drum by the end of the first one-fourth of the specified mixing time.

(*d*) When a stationary mixer is used for partial mixing of the concrete (shrink-mixing), the mixing time in the stationary mixer may be reduced to the minimum required (about 30 sec.) to intermingle the ingredients.

(*e*) When a truck mixer is used either for complete mixing or to finish the partial mixing done in a stationary mixer, each batch of concrete shall be mixed not less than 50 nor more than 100 revolutions of the drum or blades at the rate of rotation designated by the manufacturer of the equipment as mixing speed. Additional mixing, if any, shall be at the speed designated by the manufacturer of the equipment as agitating speed.

(*f*) When a truck mixer or truck agitator is used for transporting concrete which has been completely mixed in a stationary mixer, mixing during transportation shall be at the speed designated by the manufacturer of the equipment as agitating speed.

(*g*) Concrete shall be delivered to the site of the work and discharge from the truck mixer or agitator shall be completed within 1½ hr. after the introduction of the mixing water to the cement and aggregates, or the cement to the aggregates, unless a longer time is specifically authorized by the purchaser. In hot weather, or under conditions contributing to quick stiffening of the concrete, a time less than 1½ hr. may be specified by the purchaser.

(*h*) Concrete delivered in outdoor temperatures lower than 40 F. shall

arrive at the work having a temperature not less than 60 F. nor greater than 90 F., unless otherwise specified or permitted by the purchaser.

Inspection

10. Proper facilities shall be provided for the purchaser to inspect ingredients and processes used in the manufacture and delivery of the concrete. The manufacturer shall afford the inspector representing the purchaser all reasonable facilities, without charge, for securing samples to determine whether the concrete is being furnished in accordance with these specifications. All tests and inspections shall be so conducted as not to interfere unnecessarily with the manufacture and delivery of the concrete.

Sampling

11. Samples of concrete shall be obtained in accordance with the Standard Method of Sampling Fresh Concrete (A.S.T.M. Designation: C 172),[3] except in the case of individual samples secured to determine uniformity of consistency. In securing individual samples to determine uniformity of consistency as provided in Section 8 (*d*), the A.S.T.M. Method C 172 shall be followed, but the requirements shall be so modified as to permit of obtaining two samples, one at the one-quarter point and one at the three-quarter point of the load.

Slump

12. Slump tests shall be made at the option of the purchaser. If the measured slump falls outside the limits specified, a check test shall be made. In the event of a second failure the purchaser may refuse to permit the use of the load of concrete represented.

Certification

13. The purchaser may require the manufacturer of the ready-mixed concrete to furnish, with each load, a statement showing the proportions used in making the concrete. In any event, the manufacturer shall inform the purchaser of any change in proportions.

Strength

14. (*a*) When strength is used as a basis for acceptance of concrete made in accordance with these specifications, standard tests (see Section 17) shall be made frequently by the purchaser and, in general, not less frequently than one strength test for each fifty loads of each class of concrete, except that in no case shall a given class of concrete be represented by less than three tests.

(*b*) For a *strength test* three standard test specimens shall be made from a composite sample secured as required in Section 11. The test result shall be the average of the strengths of the three specimens, except that, if one specimen in a test shows manifest evidence of improper sampling, molding, or testing, it shall be discarded and the remaining two strengths averaged. Should more than one specimen representing a given test show definite defects, the entire test shall be discarded.

(*c*) The representative of the purchaser shall ascertain and record the exact location in the work at which each load represented by a strength test is deposited.

(*d*) To conform to the requirements of these specifications, the average of all of the strength tests representing each class of concrete as well as the average of any five consecutive strength tests representing each class of concrete shall be equal to or greater than the specified strength and no strength test shall have an average value less than 80 per cent of the specified strength.

Alternate Strength Basis

15. The provisions of Section 14 may be waived, at the option of the purchaser,

if the manufacturer can furnish evidence satisfactory to the purchaser that concrete of the proportions and made with the materials which he proposes to use will have the specified strength.

Failure to Meet Specifications

16. In the event that the concrete fails to meet the requirements of these specifications in any respect, the manufacturer of the ready-mixed concrete and the purchaser shall confer to determine if agreement can be reached as to what adjustment, if any, shall be made. If an agreement on a mutually satisfactory adjustment cannot be reached by the manufacturer and the purchaser, a decision shall be made by a panel of three qualified engineers, one of whom shall be designated by the purchaser, one by the manufacturer, and the third chosen by these two members of the panel. The panel shall use such means as it considers appropriate to determine the quality of the concrete, including, if deemed advisable by it, chemical analyses, strength tests of cores cut from the structure, and load tests of the finished structure. The question of responsibility for the cost of such arbitration shall be determined by the panel. Its decision shall be binding except as modified by a court decision.

Methods of Testing

17. Methods of testing ready-mixed concrete shall be in accordance with the following methods of the America Society for Testing Materials:

(*a*) *Slump.*—Standard Method of Slump Test for Consistency of Portland-Cement Concrete (A.S.T.M. Designation: C 143).[3]

(*b*) *Compression Test Specimens.*—Standard Method of Making and Curing Concrete Compression and Flexure Test Specimens in the Field (A.S.T.M. Designation: C 31),[3] except that Section 7 (*c*) of Method C 31 shall not apply to specimens used as a basis for acceptance.

(*c*) *Compression Tests.*—Standard Method of Test for Compressive Strength of Molded Concrete Cylinders (A.S.T.M. Designation: C 39).[3]

(*d*) *Flexure Tests.*—Standard Method of Test for Flexural Strength of Concrete (Laboratory Method Using Simple Beam with Third-Point Loading) (A.S.T.M. Designation: C 78).[3]

(*e*) *Core Tests.*—Standard Methods of Securing, Preparing, and Testing Specimens from Hardened Concrete for Compressive and Flexural Strengths (A.S.T.M. Designation: C 42).[3]

(*f*) *Yield.*—Standard Method of Test for Weight per Cubic Foot, Yield, and Air Content (Gravimetric) of Concrete (A.S.T.M. Designation: C 138).[3]

(*g*) *Cement Content of Hardened Concrete.*—Standard Method of Test for Cement Content of Hardened Portland-Cement Concrete (A.S.T.M. Designation: C 85).[3]

Standard Methods of

SECURING, PREPARING, AND TESTING SPECIMENS FROM HARDENED CONCRETE FOR COMPRESSIVE AND FLEXURAL STRENGTHS[1]



A.S.T.M. Designation: C 42 - 44

ADOPTED, 1927; REVISED, 1931, 1939, 1942, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 42; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods cover the procedure for securing, preparing, and testing specimens of hardened concrete from structures and pavements.

Precautions

2. A specimen to be tested for strength shall not be removed from the structure until the concrete has become hard enough to permit its removal without disturbing the bond between the mortar and the coarse aggregate. In general, the concrete shall be 14 days old before the specimens are removed. Specimens that show abnormal defects or that have been damaged in removal shall not be used.

Apparatus

3. (*a*) *Core Drill.*—A core drill shall be used for securing cylindrical core specimens. For specimens taken perpendicular to a horizontal surface, a shot drill is satisfactory; for specimens taken perpendicular to a vertical surface, a diamond drill shall be used.

(*b*) *Saw.*—A saw shall be used for securing beam specimens from the structure or pavement for flexural strength tests. The saw shall have a diamond or silicon carbide cutting edge and shall have adjustments that will permit cutting specimens which conform to the dimensions prescribed in Section 4(*b*).

Test Specimens

4. (*a*) *Core Specimens.*—A core specimen for the determination of pavement thickness shall have a diameter of at least 4 in. A core specimen for the determination of compressive strength shall have a diameter at least three times the maximum nominal size of the coarse aggregate used in the concrete, and in no case shall the diameter of the specimen be less than twice the maximum nominal size of the coarse aggregate. The length of the specimen, when capped, shall be as nearly as practicable twice its diameter.

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates.

[2] Prior to adoption as standard these methods were published as tentative from 1921 to 1927, being revised in 1925 and 1927.

(*b*) *Beam Specimens.*—A beam specimen for the determination of flexural strength, in general, shall have a cross-section of 6 by 6 in. (Note). The specimen shall be at least 21 in. in length, but when two tests for flexural strength are desired for one beam specimen it shall be at least 33 in. in length.

NOTE.—In many cases, particularly with prisms cut from pavement slabs, the width will be governed by the size of the coarse aggregate and the depth by the thickness of the slab.

Procedure

5. (*a*) *Core Drilling.*—A core specimen taken perpendicular to a horizontal surface shall be located, when possible, so that its axis is perpendicular to the bed of the concrete as originally placed. A specimen taken perpendicular to a vertical surface, or perpendicular to a surface with a batter, shall be taken from near the middle of a unit of deposit.

(*b*) *Slab Removal.*—A sufficiently large slab shall be removed so that the desired test specimens may be secured without the inclusion of any concrete which has been cracked, spalled, undercut, or otherwise damaged.

(*c*) *Beam Sawing.*—The sawing operation shall be so performed that the concrete will not be weakened by shock or by heating. The sawed surfaces shall be smooth, plane, parallel, and free from steps, ridges and grooves. Care shall be taken in handling sawed beam specimens to avoid chipping or cracking.

Length of Drilled Core Specimens

6. The lengths of core specimens for determining the thickness of the pavement shall be measured as prescribed in the Standard Method of Measuring Length of Drilled Concrete Cores (A.S.T.M. Designation: C 174).[3]

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Compressive Strength

7. (*a*) *End Preparation.*—Core specimens to be tested in compression shall have ends that are essentially smooth, perpendicular to the axis, and of the same diameter as the body of the specimen. The ends of specimens which have projections of $\frac{1}{4}$ in. or more above the normal plane, or which depart from perpendicularity to the axis by more than 5 deg., or whose diameter departs from the mean by more than $\frac{1}{8}$ in. shall be sawed or tooled until they conform to these tolerances.

(*b*) *Moisture Conditioning.*—Test specimens shall be completely submerged in water at room temperature for 40 to 48 hr. immediately prior to the compression test. Specimens shall be tested promptly after removal from water storage. During the period between removal from the water storage and testing, the specimens shall be kept moist by covering with a wet burlap or blanket. They shall be tested in a moist condition.

(*c*) *Capping.*—Before making the compression test, the ends of the specimen shall be capped or ground. The capped or ground surface shall be at right angles to the axis of the specimen and shall not depart from a plane by more than 0.001 in. Caps shall be made as thin as practicable and shall not flow or fracture when the specimen is tested.

NOTE.—Neat portland or alumina cement and suitable mixtures of sulfur with granular materials are recognized as suitable for capping hardened concrete specimens. Sulfur caps should be allowed to harden for at least 1 hr. before applying load; cement caps for a sufficient period to comply with the requirements prescribed in Paragraph (*c*).

(*d*) *Measurement.*—Prior to testing, the length of the capped specimen shall be measured to the nearest 0.1 in. and its average diameter determined to the nearest 0.1 in. from two measurements taken at right angles near the center of the length.

(*e*) *Testing.*—Specimens shall be tested as prescribed in Section 4 of the Standard Method of Test for Compressive Strength of Molded Concrete Cylinders (A.S.T.M. Designation: C 39).[3]

(*f*) *Calculation and Report.*—The direction of the application of the load with reference to direction of compaction of the concrete in the structure shall be reported. The compressive strength of each specimen shall be calculated in pounds per square inch based on the average diameter of the specimen. If the ratio of length to diameter of a specimen is appreciably less than two, allowance for the ratio of length to diameter shall be made by multiplying the compressive strength by the applicable correction factor given in the following table. Values not given in the table shall be determined by interpolation:

Ratio of Length of Cylinder to Diameter $\left(\frac{l}{d}\right)$	Strength Correction Factor
1.75	0.98
1.50	0.96
1.25	0.94
1.10	0.90
1.00	0.85
0.75	0.70
0.50	0.50

Flexural Strength

8. (*a*) *Moisture Conditioning.*—Test specimens shall be completely submerged in water at room temperature for 40 to 48 hr. immediately prior to the flexure test. Specimens shall be tested promptly after removal from water storage. During the period between removal from the water storage and testing, the specimens shall be kept moist by covering with a wet burlap or blanket. They shall be tested in a moist condition.

(*b*) *Testing.*—Specimens shall be tested as prescribed in the Standard Method of Test for Flexural Strength of Concrete (Using Simple Beam with Third-Point Loading) (A.S.T.M. Designation: C 78).[3]

NOTE.—The compressive strengths of portions of beams broken in flexure may be determined by testing such portions as modified cubes using the procedure prescribed in the Standard Method of Test for Compressive Strength of Concrete Using Portions of Beams Broken in Flexure (Modified Cube Method) (A.S.T.M. Designation: C 116).[3]

Standard Method of

SAMPLING FRESH CONCRETE[1]

A.S.T.M. Designation: C 172 - 44

ADOPTED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 172; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method covers the procedure for obtaining samples of fresh concrete from mixers, hoppers, or transportation units.

Size of Sample

2. The sample shall be representative of the batch. Where feasible, it shall consist of portions from different points in the batch. The composite sample shall consist of not less than 1 cu. ft. when the sample is to be used for acceptance tests. Smaller samples may be permitted for routine slump tests.

Procedure for Sampling

3. (*a*) *From Mixers Used in Construction, Other than Paving or Truck Mixers.*—The sample shall be obtained by passing a receptacle or receptacles through the discharge stream of the mixer at about the middle of the batch. Care shall be taken not to restrict the flow from the mixer in such a manner as to cause the concrete to segregate.

(*b*) *From Paving Mixers.*—The contents of the paving mixer shall be discharged upon the subgrade and the sample shall be collected from a sufficient number of points in the batch to be representative.

(*c*) *From Revolving Drum Truck Mixers or Agitators.*—The sample shall be taken in three or more regular increments throughout the discharge of the entire batch. To permit sampling, the rate of discharge of the batch shall be regulated by the rate of revolution of the drum and not by the size of the gate opening.

(*d*) *From Open-Top Truck Mixers and Agitators.*—The sample shall be taken directly from the mixer and shall consist of portions from not less than three points in the batch.

(*e*) *From Receiving Hoppers, Buckets, etc.*—Samples shall be taken by whichever of the procedures described in Paragraphs (*a*), (*b*), or (*c*) is most applicable under the conditions.

Mixing Composite Sample

4. The composite sample shall be mixed with a shovel sufficiently to insure homogeneity and immediately molded into test specimens.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates.

[2] Prior to adoption as standard, this method was published as tentative from 1942 to 1944.

Standard Method of Test for

CEMENT CONTENT OF HARDENED PORTLAND-CEMENT CONCRETE[1]

A.S.T.M. Designation: C 85 - 42

ADOPTED, 1936; REVISED, 1942.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 85; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test for determining the cement content of concrete is applicable to hardened portland-cement concretes *except those containing certain aggregates or admixtures which liberate soluble silica under the conditions of the test, such as slag, diatomites, and sodium silicate.*

Reagents

2. (*a*) *Hydrochloric Acid (approximately 3.3 N).*—Add 200 ml. of HCl (sp. gr. 1.19) to 600 ml. of distilled water.

(*b*) *Sodium Hydroxide (approximately 1 N).*—Dissolve 20 g. of NaOH in 200 ml. of water and dilute to a volume of 500 ml.

(*c*) *Hydrofluoric Acid (40 per cent).*

(*d*) *Sulfuric Acid (sp. gr. 1.84).*

Preparation of Sample

3. Every precaution shall be taken to have the sample of concrete used for analysis truly representative of the material under consideration. Several portions weighing at least 10 lb. each shall be taken to avoid all but slight inequalities of the concrete mix. These portions shall then be broken up, crushed in a suitable machine to about $\frac{1}{2}$ in. in size, and reduced to a fineness of approximately 150 to 200-mesh in a ball mill, disk pulverizer, or other device. Care shall be taken that the finer fractions of the broken sample, which are richer in cement, are not discarded or lost. After thorough mixing and quartering, a portion approximating 100 g. shall be taken and carefully freed, by means of a strong magnet, from particles of metallic iron abraded from the pulverizer or ball mill. The clean sample then shall be dried at 105 C. for at least 2 hr.

Procedure

4. (*a*) Weigh into each of three 250-ml. beakers, not less than a 2-g. portion

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates.

[2] Prior to adoption as standard, this method was published as tentative from 1931 to 1936.

of the prepared sample. Moisten with a stream of hot water, while stirring to prevent adhesion to the beaker or the formation of lumps in the mass. Slowly add 100 ml. of 3.3 *N* HCl and stir thoroughly. The lumps which tend to form should be reduced with the glass rod. After the evolution of CO_2 has ceased and the reaction is apparently complete, heat gently for a few minutes and allow the contents of the beaker to settle. Decant through an ignited and weighed Gooch crucible which contains a mat of short asbestos shreds, practically insoluble in HCl, thick enough to be opaque to light. Once the filtration has begun, care shall be taken so that the mat and accumulated residue do not dry out completely until the filtration process is complete. Regulate the suction so as to maintain a rapid rate of dropping during the greater part of the filtration. Retain as much of the residue in the beaker as possible. Wash by decantation twice with hot water. Add 75 ml. of 1 *N* NaOH to the residue while stirring and heat to about 75 C. Decant as before and wash twice with hot water. Transfer the residue to the crucible and wash with at least 60 ml. of hot water.

(*b*) The filtrate now contains the silica in the form of silicic acid either in true solution or in suspension in the hydrochloric acid medium. If the aggregates of the original sample are largely calcareous or dolomitic, add 10 ml. of HCl (sp. gr. 1.19) to the solution. Transfer to a suitable beaker, with several rinsings of the filter flask. Evaporate to dryness with great care to minimize spattering, bake at not over 120 C. for 1 hr., moisten with HCl, (sp. gr. 1.19), evaporate and bake again, and take up for filtration in 75 ml. of 2 to 3 *N* HCl heated to boiling. Filter through an ashless filter-paper, and wash the residue with 50 ml. of hot 1 *N* HCl and then with hot water until the washings are free from chlorides. Repeat the evaporation and filtering processes to recover the small amounts of silica dissolved and add these to the first residue. Determine the silica present in the sample by treatment with HF and H_2SO_4 in accordance with the procedure given in Section 8 of the Standard Methods of Chemical Analysis of Portland Cement (A.S.T.M. Designation: C 114) of the American Society for Testing Materials.[3]

Correction Factor

5. When the aggregates are available that were used in the concrete being analyzed, a blank test shall be run on these aggregates to determine their content of silica, soluble under the conditions of the test (Section 4). This content of soluble silica shall then be used as a correction factor and be subtracted from the total soluble silica found in the concrete, the difference being due to the cement contained in the specimen.

Calculation

6. The percentage of cement in the sample shall be calculated by dividing the percentage of silica found by the factor 0.2175,[4] provided it is not known that the silica content of the cement differs from this value. When possible, the *known value* shall be taken as the factor.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

[4] For the correctness of this factor as an average silica content of American cements, see Report of Subcommittee I on Definitions and Chemical Limitations, of Committee C-1 on Cement, *Proceedings*, Am. Soc. Testing Mats., Vol. 28, Part I, pp. 238 to 243 (1928).

AMERICAN ASSOCIATION STATE
HIGHWAY OFFICIALS STANDARD
A.A.S.H.O. NO.: T 23

Standard Method of

MAKING AND CURING CONCRETE COMPRESSION AND FLEXURE TEST SPECIMENS IN THE FIELD[1]

A.S.T.M. Designation: C 31 – 44

ADOPTED, 1921; REVISED, 1927, 1931, 1933, 1938, 1939, 1942, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 31; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method covers the procedure for making and curing compression and flexure test specimens of concrete sampled from concrete being used in construction.

NOTE.—For the method of making and curing concrete specimens in the laboratory see the Tentative Method of Making and Curing Concrete Compression and Flexure Test Specimens in the Laboratory (A.S.T.M. Designation: C 192).[3]

Sampling of Concrete

2. Samples of concrete for test specimens shall be taken in accordance with the Standard Method of Sampling Fresh Concrete (A.S.T.M. Designation: C 172).[3] The location in the work of the batch of concrete thus sampled shall be noted for further reference.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates.

[2] Prior to adoption as standard, this method was published as tentative from 1920 to 1921.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

COMPRESSION TEST SPECIMENS

Size of Specimens

3. Compression test specimens shall be cylindrical with a length equal to twice the diameter. Standard cylindrical specimens shall be 6 in. in diameter by 12 in. in length if the coarse aggregate does not exceed 2 in. in nominal size. Smaller test specimens shall have a ratio of diameter of specimen to maximum size aggregate of not less than 3 to 1, except that the diameter of the specimen shall not be less than 3 in. for mixtures containing aggregate more than 5 per cent of which is retained on a No. 4 (4760-micron) sieve. For concrete containing aggregate larger than 2 in., the cylindrical specimens shall have a diameter at least three times the maximum nominal size of aggregate. The oversize of any nominal size aggregate used shall not exceed the requirements prescribed in the Standard Specifications for Concrete Aggregates (A.S.T.M. Designation: C 33).[3]

Molds

4. Molds for compression test specimens shall be cylindrical in form, made of

nonabsorbent material, and shall be substantial enough to hold their form during the molding of test specimens. They shall not vary from the standard diameter by more than $\frac{1}{16}$ in. nor from the standard length by more than $\frac{1}{4}$ in. Metal molds shall each be provided with a machined metal base plate and means shall be provided for securing the base plate to the mold. The assembled mold and base plate shall be watertight and shall be oiled with mineral oil before use. Molds made of material other than metal shall be provided with a base plate or bottom. Assembled molds of any type shall be watertight.

NOTE.—Satisfactory molds may be made from cold-drawn, seamless steel tubing or from steel pipe machined on the inside. These tubular sections shall be cut to the proper length, split along one element and fitted with a circumferential metal band and bolt for closing. Satisfactory molds may also be made from iron or steel castings. In general, molds made from formed sheet metal are not satisfactory. Paraffined cardboard molds will give good results under expert supervision.

Molding Specimens

5. The test specimens shall be formed by placing the concrete in the mold in three layers of approximately equal volume. In placing each scoopful of concrete, the scoop shall be moved around the top edge of the mold as the concrete slides from it in order to insure a symmetrical distribution of the concrete within the mold. The concrete shall be further distributed by a circular motion of the tamping rod. Each layer shall be rodded with 25 strokes of a tamping rod $\frac{5}{8}$ in. in diameter and approximately 24 in. in length, bullet-shaped at the lower end. The strokes shall be distributed uniformly over the cross-section of the mold and shall penetrate into the underlying layer. The bottom layer shall be rodded throughout its depth. After the top layer has been rodded, the surface of the concrete shall be struck off with a trowel and covered with a glass or metal plate to prevent evaporation.

Capping Specimens

6. (*a*) If metal molds are used, the test specimens may be capped with a thin layer of stiff, neat-cement paste after the concrete has ceased settling in the molds, generally from 2 to 4 hr. or more after molding. The cap shall be formed by means of a piece of plate glass not less than $\frac{1}{4}$ in. in thickness or a machined metal plate not less than $\frac{1}{2}$ in. in thickness and having a minimum surface dimension at least 1 in. larger than the diameter of the mold. The plate shall be worked on the cement paste until its lower surface rests on top of the mold. The cement for capping shall be mixed to a stiff paste 2 to 4 hr. before it is to be used in order to avoid the tendency of the cap to shrink. Adhesion of the concrete to the top and bottom plates may be avoided by coating them with oil or grease.

(*b*) Specimens not capped with neat-cement paste as described in Paragraph (*a*) shall be ground or capped before testing. In all cases the capped or ground surface shall not depart from a plane by more than 0.001 in. and shall be at right angles to the axis of the specimen. Caps shall be made as thin as practicable and shall not flow or fracture when the specimen is tested.

NOTE.—Neat portland or alumina cement and suitable mixtures of sulfur with granular materials are recognized as suitable for capping hardened concrete specimens. Sulfur caps should be allowed to harden for at least 1 hr. before applying load; cement caps for a sufficient period to comply with the requirements prescribed in Paragraph (*b*).

Curing Specimens

7. (*a*) During the first 24 hr. all test specimens shall be kept in a storage box (Note 1) so constructed and located on the

work that its air temperature when containing concrete specimens will remain within 60 to 80 F. (16 to 27C.), or other suitable means shall be used that provide similar temperature conditions.

NOTE 1.—It is suggested that the storage box be made of 1-in. dressed tongue-and-groove lumber, well braced with battens to avoid warping. The box should be well painted inside and outside and should be provided with a hinged cover and padlock.

(*b*) Test specimens made to check the adequacy of the laboratory design for strength of the concrete, or as the basis for acceptance, shall be removed from the molds at the end of 24 hr. and stored in a moist condition (Note 2) at a temperature within the range of 65 to 75 F. (18 to 24 C.) (Note 3) until the time of test. Specimens shall not be exposed to a stream of running water. If storage in water is desired, a saturated lime solution shall be used.

NOTE 2.—Moist condition is that in which free water is maintained on the surfaces of the specimens at all times.

NOTE 3.—Attention is directed to the fact that the temperature within damp sand and under wet burlap or similar materials will always be lower than the temperature in the surrounding atmosphere if evaporation takes place.

(*c*) Test specimens for determining when a structure may be put into service shall be removed from the molds at the end of 24 hr. and stored in the structure as near to the point of sampling as possible and shall receive, in so far as practicable, the same protection from the elements on all surfaces as is given to the portions of the structure which they represent. Field control specimens shall be protected from injury while on the work. For 28-day tests the specimens shall be sent to the laboratory not more than 7 days prior to the time of test. For other periods of test the specimens shall be kept in the field at least three fourths of the test period. While in the laboratory the specimens shall be kept at laboratory temperature until 24 to 48 hr. before testing, during which final period they shall be immersed in water at laboratory temperature.

FLEXURE TEST SPECIMENS

Size of Specimens

8. The cross-section of the flexure test specimen shall be 6 by 6 in. if the coarse aggregate does not exceed 2 in. in nominal size. For larger coarse aggregate, the minimum cross-sectional dimension shall be not less than three times the maximum nominal size of the coarse aggregate. The oversize of any nominal size used shall not exceed the requirements prescribed in the Standard Specifications for Concrete Aggregates (A.S.T.M. Designation: C 33).[3]

Molds

9. Molds for flexure test specimens shall be rigid and nonabsorptive and shall be at least 3 in. longer than the required span length as prescribed in Section 3 of the Standard Method of Test for Flexural Strength of Concrete (Using Simple Beam with Third-Point Loading) (A.S.T.M. Designation: C 78).[3] Means shall be provided for securing the base plate to the mold. The assembled mold and base plate shall be watertight and shall be lightly oiled with a mineral oil before use.

Molding Specimens

10. The test specimen shall be formed with its long axis horizontal. The concrete shall be placed in layers approximately 3 in. in depth and each layer shall be rodded 50 times for each square foot of area. The top layer shall slightly overfill the mold. After each layer is rodded, the concrete shall be spaded along the sides and ends with a mason's trowel or other suitable tool. When the rodding

and spading operations are completed, the top shall be struck off with a straightedge and finished with a wood float. The test specimen shall be made promptly and without interruption.

Curing Specimens

11. (*a*) Test specimens made to check the adequacy of the laboratory design for strength of the concrete, or as the basis for acceptance, shall be covered immediately after molding with a double layer of wet burlap which shall be kept wet until the specimens are removed from the molds. During the first 24 hr. the specimens shall be cured under the conditions specified in Section 7 (*a*). At the end of the 24-hr. period the specimens shall be removed from the molds and stored in a moist condition as specified in Section 7 (*b*).

(*b*) Test specimens for determining when a structure may be put into service, shall be cured as nearly as practicable, in the same manner as the concrete in the structure. At the end of 24 hr. the specimens shall be taken in the molds to a location preferably near a field laboratory, removed from the molds and stored by placing them on the ground as molded, with their top surfaces up. The sides and ends of the specimens shall then be banked with damp earth or sand which shall be kept damp, leaving the top surfaces exposed to the specified curing treatment. At the end of the curing period the specimens shall be left in place with the top surfaces exposed to the weather in the same manner as the structure. Specimens shall be tested in the moist condition resulting from the specified curing treatment.

Standard Method of Test for

COMPRESSIVE STRENGTH OF MOLDED CONCRETE CYLINDERS[1]

A.S.T.M. Designation: C 39 - 44

ADOPTED, 1925; REVISED, 1927, 1933, 1939, 1942, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 39; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method covers the procedure for compression tests of molded concrete cylinders.

NOTE.—For methods of molding concrete specimens see the Tentative Method of Making and Curing Concrete Compression and Flexure Specimens in the Laboratory (A.S.T.M. Designation: C 192),[3] and the Standard Method of Making and Curing Concrete Compression and Flexure Test Specimens in the Field (A.S.T.M. Designation: C 31).[3]

Apparatus

2. The testing machine may be of any type of sufficient capacity which will provide the rate of loading prescribed in Section 4 (*b*). It shall conform to the requirements of Section 27 of the Standard Methods of Verification of Testing Machines (A.S.T.M. Designation: E 4).[3] The testing machine shall be equipped with two steel bearing blocks with hardened faces (Note) one of which is a spherically seated block that normally will bear on the upper surface of the specimen, and the other a plain rigid block on which the specimen will rest. The bearing faces shall be at least as large and preferably slightly larger than the surface of the specimen to which the load is applied. The bearing faces, when new, shall not depart from a plane by more than 0.0005 in. at any point, and they shall be maintained within a permissible variation limit of 0.001 in. In the spherically seated block the diameter of the sphere shall not greatly exceed the diameter of the specimen and the center of the sphere shall coincide with the center of the bearing face. The movable portion of this block shall be held closely in the spherical seat, but the design shall be such that the bearing face can be rotated freely and tilted through small angles in any direction.

NOTE.—It is desirable that the bearing faces of blocks used for compression testing of concrete have a Rockwell hardness of not less than C 60.

Test Specimens

3. Compression tests of moist cured specimens shall be made as soon as practicable after removal from the curing room. Test specimens, during the pe-

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates.

[2] Prior to adoption as standard, this method was published as tentative from 1921 to 1925.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

riod between their removal from the moist room and testing shall be kept moist by a wet burlap or blanket covering. They shall be tested in a moist condition. The diameter of the test specimen shall be determined to the nearest 0.01 in. by averaging two diameters measured at right angles to each other near the center of the length of the specimen. This average diameter shall be used for calculating the cross-sectional area. The length of the specimen including caps shall be measured to the nearest 0.1 in.

Procedure

4. (*a*) *Placing the Specimen.*—The plain (lower) bearing block shall be placed, with its hardened face up, on the table or platen of the testing machine directly under the spherically seated (upper) bearing block. The bearing face shall be wiped clean and the test specimen placed on it. The axis of the specimen shall be carefully aligned with the center of thrust of the spherically seated block. As the spherically seated block is brought to bear on the specimen its movable portion shall be rotated gently by hand so that uniform seating is obtained.

(*b*) *Rate of Loading.*—The load shall be applied continuously and without shock. In testing machines of the screw type the moving head shall travel at a rate of about 0.05 in. per min. when the machine is running idle. In hydraulically operated machines the load shall be applied at a constant rate within the range 20 to 50 psi. per sec. During the application of the first half of the maximum load a higher rate of loading shall be permitted.

(*c*) The load shall be increased until the specimen fails and the maximum load carried by the specimen during the test shall be recorded. The type of failure and the appearance of the concrete shall be noted.

Calculation

5. The compressive strength of the specimen shall be calculated by dividing the maximum load carried by the specimen during the test by the average cross-sectional area determined as described in Section 3, and shall be expressed to the nearest 10 psi.

Report

6. The report shall include the following:

(*1*) Identification number,

(*2*) Diameter (and length, if not standard), in inches,

(*3*) Cross-sectional area, in square inches,

(*4*) Maximum load, in pounds,

(*5*) Compressive strength calculated to the nearest 10 psi.,

(*6*) Type of fracture, if other than the usual cone,

(*7*) Defects in either specimen or caps,

(*8*) Age of specimen.

Standard Method of Test for

COMPRESSIVE STRENGTH OF CONCRETE USING PORTIONS OF BEAMS BROKEN IN FLEXURE[1]

MODIFIED CUBE METHOD

A.S.T.M. Designation: C 116 - 44

ADOPTED, 1936; REVISED, 1939, 1942, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 116; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test covers the procedure for determining the compressive strength of concrete, using for the test specimens portions of beams broken in flexure.

NOTE.—For the method of making the flexural strength test, see the Standard Method of Test for Flexural Strength of Concrete (Using Simple Beam with Third-Point Loading) (A.S.T.M. Designation: C 78).[3]

Test Specimens

2. The broken portions of beams selected for the compression test shall have a length not less than 2 in. greater than the depth of the beam and shall be free from cracks, chipped surfaces, or other obvious defects.

Preparation of Test Specimen

3. (*a*) The top and bottom of the beam shall be ground or capped in such a manner that plane surfaces are secured. The caps shall cover the full width of the beam and shall be of such length as to permit the adjustment of the bearing plates for the test so that the upper bearing plate may be placed directly over the lower bearing plate. The material used for capping and the thickness of the cap shall be such that it will not flow or fracture under the load.

NOTE.—Neat portland or alumina cement and suitable mixtures of sulfur with granular materials are recognized as suitable for capping hardened concrete specimens. Sulfur caps should be allowed to harden for at least 1 hr. before applying load; cement caps for a sufficient period to comply with the requirements prescribed in Paragraph (*a*).

(*b*) During the interval between testing of specimens as beams and testing the broken portions as cubes, the specimens shall be kept moist by storage in a moist closet or under wet burlap.

Procedure

4. (*a*) Only the ultimate compressive

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates.

[2] Prior to adoption as standard, this method was published as tentative from 1935 to 1936.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

strength shall be determined. The specimen shall be tested between machined metal bearing plates not less than $\frac{3}{4}$ in. in thickness. The diameter of the spherical bearing block shall be at least 75 per cent of the depth of the beam. The plates shall be of such dimensions that the contact faces shall be substantially square and shall have the same dimensions as the nominal width of the beam tested. The upper bearing plate shall be placed directly over the lower plate. A device that insures the proper location of the upper plate with reference to the lower plate is shown in Fig. 1. The load shall be applied to the bearing blocks by means of an adjustable head.

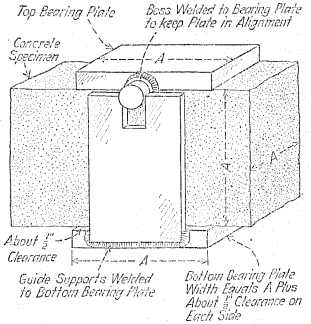


FIG. 1.—Device for Aligning Bearing Plates.

(*b*) *Application of Load.*—The load shall be applied without shock. The moving head of the screw type of testing machine shall travel at the rate of about 0.05 in. per min. when the machine is running idle. In hydraulic machines the loading shall be adjusted to a constant rate within the limits of 35 ± 15 psi. per sec.

Calculation and Report

5. The total load indicated by the testing machine at failure of the test specimen shall be recorded and the unit compressive strength calculated in pounds per square inch, taking as the cross-sectional area the average of the upper and lower contact areas. The type of failure and appearance of the concrete shall be noted.

Standard Method of Test for

FLOW OF PORTLAND-CEMENT CONCRETE BY USE OF THE FLOW TABLE[1]

A.S.T.M. Designation: C 124 – 39

ADOPTED, 1938; REVISED, 1939.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 124; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method is intended for determining the flow of concrete.

Apparatus

2. (*a*) *Mold.*—The mold shall be made of a smooth metal casting in the form of the frustum of a cone with a base 10 in. in diameter, upper surface $6\frac{3}{4}$ in. in diameter, and altitude 5 in.; the base and the top shall be open and at right angles to the axis of the cone. The mold shall be provided with handles. (See Fig. 1.)

(*b*) *Flow Table.*—The flow table shall conform to the design shown in Fig. 2 and shall be mounted on and bolted to a concrete base having a height of 15 to 20 in. and weighing not less than 300 lb.

Sample

3. Samples of concrete for test specimens shall be taken at the mixer, or in the case of ready-mixed concrete, from the transportation vehicle during discharge. The sample of concrete from which test specimens are made shall be representative of the entire batch. Such samples shall be obtained by repeatedly passing a scoop or pail through the discharging stream of concrete, starting the sampling operation at the beginning of discharge, and repeating the operation until the entire batch is discharged. The sample thus obtained shall be transported to the place of molding of the specimen, and to counteract segregation the concrete shall be mixed with a shovel until it is uniform in appearance. The location in the work of the batch of concrete thus sampled shall be noted for future reference. In the case of paving concrete, samples may be taken from the batch immediately after depositing on the subgrade. At least five samples shall be taken from different portions of the pile, and these samples shall be thoroughly mixed to form the test specimen.

Procedure

4. Immediately preceding the test, the table top shall be wetted and cleaned of all gritty material and the excess

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates.

[2] Prior to adoption as standard, this method was published as tentative from 1936 to 1938.

water removed with a rubber squeegee. The mold, centered on the table, shall be firmly held in place and filled in two layers, each approximately one-half the volume of the mold. Each layer shall be rodded with 25 strokes of a round, nonmetallic rod $\frac{5}{8}$ in. in diameter and 24 in. in length, having a bullet-pointed end. The strokes shall be distributed in a uniform manner over the cross-section of the mold and shall penetrate into the underlying layer. The bottom layer shall be rodded throughout its depth. After the top layer has been rodded, the surface of the concrete shall be struck off with a trowel so that the mold is exactly filled. The excess concrete which has overflowed the mold shall be removed and the area of the table outside of the mold again cleaned. The mold shall be immediately removed from the concrete by a steady, upward pull. The table shall then be raised and dropped $\frac{1}{2}$ in., 15 times in about 15 sec. The diameter of the spread concrete shall be the average of six symmetrically distributed caliper measurements read to the nearest $\frac{1}{4}$ in.

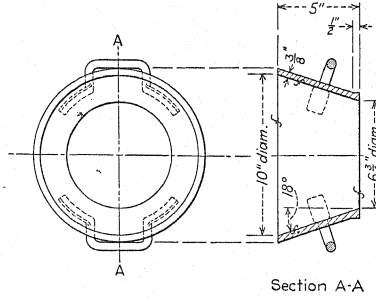


FIG. 1.—Mold for Flow Test.

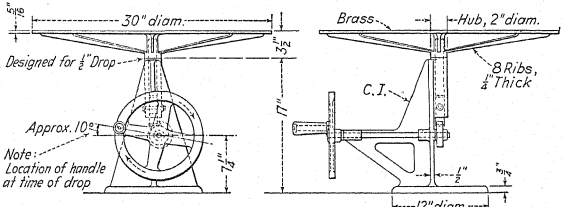


FIG 2.—Flow Table Assembly.

Flow

5. The flow of the concrete shall be recorded as the percentage increase in diameter of the spread concrete over the base diameter of the molded concrete, calculated from the following formula:

$$\text{Flow, per cent} = \frac{\text{spread diameter} - 10 \text{ in.}}{10} \times 100$$

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A.A.S.H.O. NO.: T 119-42

Standard Method of

SLUMP TEST FOR CONSISTENCY OF PORTLAND-CEMENT CONCRETE[1]

A.S.T.M. Designation: C 143 – 39

ADOPTED, 1939.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 143; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test covers the procedure to be used both in the laboratory and in the field for determining consistency of concrete.[3]

Apparatus

2. The test specimen shall be formed in a mold of No. 16 gage galvanized metal in the form of the lateral surface of the frustrum of a cone with the base 8 in. in diameter, the top 4 in. in diameter, and the altitude 12 in. The base and the top shall be open and parallel to each other and at right angles to the axis of the cone. The mold shall be provided with foot pieces and handles as shown in Fig. 1.

Sample

3. Samples of concrete for test specimens shall be taken at the mixer or, in the case of ready-mixed concrete, from the transportation vehicle during discharge. The sample of concrete from which test specimens are made shall be representative of the entire batch. Such samples shall be obtained by repeatedly passing a scoop or pail through the discharging stream of concrete, starting the sampling operation at the beginning of discharge and repeating the operation until the entire batch is discharged. The sample thus obtained shall be transported to the place of molding of the specimen, and to counteract segregation the concrete shall be mixed with a shovel until it is uniform in appearance. The location in the work of the batch of concrete thus sampled shall be noted for future reference. In the case of paving concrete, samples may be taken from the batch immediately after depositing on the subgrade. At least five samples shall be taken from different portions of the pile and these samples shall be

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates.

[2] Prior to adoption as standard, this method was published as tentative from 1922 to 1939, being revised in 1925, 1926, 1932, and 1939. This method was formerly published under the jurisdiction of A.S.T.M. Committee D-4 on Road and Paving Materials and was designated D 138; but it was transferred in 1939 to Committee C-9 and redesignated C 143.

[3] This test is not considered applicable when there is a considerable amount of coarse aggregate over 2 in. in size in the concrete. The committee is now working on a method suitable for determining the consistency of concrete using aggregate over 2 in. in size.

thoroughly mixed to form the test specimen.

Procedure

4. The mold shall be dampened and placed on a flat, moist, nonabsorbent surface. From the sample of concrete obtained as described in Section 3, the mold shall immediately be filled in three layers, each approximately one-third the volume of the mold. In placing each scoopful of concrete the scoop shall be moved around the top edge of the mold as the concrete slides from it, in order to insure symmetrical distribution of concrete within the mold. Each layer shall be rodded with 25 strokes of a $\frac{5}{8}$-in. rod, 24 in. in length, bullet pointed at the lower end. The strokes shall be distributed in a uniform manner over the cross-section of the mold and shall penetrate into the underlying layer. The bottom layer shall be rodded throughout its depth. After the top layer has been rodded, the surface of the concrete shall be struck off with a trowel so that the mold is exactly filled. The mold shall be immediately removed from the concrete by raising it carefully in a vertical direction. The slump shall then be measured immediately by determining the difference between the height of the mold and the height at the vertical axis of the specimen.

Slump

5. The consistency shall be recorded in terms of inches of subsidence of the specimen during the test, which shall be known as the slump:

Slump = 12 — inches of height after subsidence

NOTE.—After the slump measurement is completed, the side of the concrete frustrum

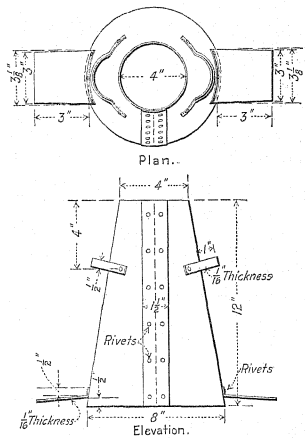


FIG. 1.—Mold for Slump Test.

should be tapped gently with the tamping rod. The behavior of the concrete under this treatment is a valuable indication of the cohesiveness, workability, and placeability of the mix. A well proportioned workable mix will gradually slump to lower elevations and retain its original identity, while a poor mix will crumble, segregate, and fall apart.

Standard Method of

MEASURING LENGTH OF DRILLED CONCRETE CORES[1]

A.S.T.M. Designation: C 174 – 44

ADOPTED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 174; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method covers the procedure for determining the length of a core drilled from a concrete structure, particularly from a concrete pavement.

Apparatus

2. (*a*) The apparatus shall be a calipering device that will measure the length of axial elements of the core. While the details of the mechanical design are not prescribed, the apparatus shall conform to the requirements of Paragraphs (*b*) to (*f*).[3]

(*b*) The apparatus shall be so designed that the specimen will be held with its axis in a vertical position by three symmetrically placed supports bearing against the lower end. These supports shall be short posts or studs of hardened steel, and the ends that bear against the surface of the specimen shall be rounded to a radius of not less than $\frac{1}{4}$ in. and not more than $\frac{1}{2}$ in.

(*c*) The apparatus shall provide for the accommodation of specimens of different nominal lengths over a range of at least 4 to 10 in.

(*d*) The calipering apparatus shall be so designed that it will be possible to make a length measurement at the center of the upper end of the specimen and at eight additional points spaced at equal intervals along the circumference of a circle whose center point coincides with that of the end area of the specimen and whose radius shall not exceed the radius of the specimen less $1\frac{1}{2}$ in.

(*e*) The measuring rod or other device that makes contact with the end surface of the specimen for measurement shall be rounded to a radius of $\frac{1}{8}$ in. The scale on which the length readings are made shall be marked with clear, definite, accurately spaced graduations. The spacing of the graduations shall be 0.10 in. or a decimal part thereof.

(*f*) The apparatus shall be stable and sufficiently rigid to maintain its shape and alignment without a distortion or deflection of more than 0.01 in. during all normal measuring operations.

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[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates.

[2] Prior to adoption as standard, this method was published as tentative from 1942 to 1944.

[3] For further information relating to the development of this method and apparatus, reference should be made to the "Project Report on a Study of Methods of Measurement of the Length of Cores Drilled from Concrete Structures," prepared by L. W. Teller for Subcommittee VII on Methods and Apparatus for Testing Concrete, of Committee C-9, see *Proceedings*, Am. Soc. Testing Mats., Vol. 42 (1942).

Test Specimens

3. Cores used as specimens for length measurement shall be in every way representative of the concrete in the structure from which they are removed. The specimen shall be drilled with the axis normal to the surface of the structure, and the ends shall be free from all conditions not typical of the surfaces of the structure. Cores that show abnormal defects or that have been damaged appreciably in the drilling operation shall not be used.

Procedure

4. (*a*) Before any measurements of the core length are made, the apparatus shall be calibrated with suitable gages so that errors caused by mechanical imperfections in the apparatus are known. When these errors exceed 0.01 in., suitable corrections shall be applied to the core length measurements.

(*b*) The specimen shall be placed in the measuring apparatus with the smooth end of the core, that is, the end that represents the upper surface of a pavement slab or a formed surface in the case of other structures, placed down so as to bear against the three hardened-steel supports. The specimen shall be so placed on the supports that the central measuring position of the measuring apparatus is directly over the mid-point of the upper end of the specimen.

(*c*) Nine measurements of the length shall be made on each specimen, one at the central position and one each at eight additional positions spaced at equal intervals along the circumference of the circle of measurement described in Section 2 (*d*). Each of these nine measurements shall be read directly to tenths of inches and to hundredths of inches either directly or by estimation.

NOTE—If, in the course of the measuring operation, it is discovered that at one or more of the measuring points the surface of the specimen is not representative of the general plane of the core end because of a small projection or depression, the specimen shall be rotated slightly about its axis and a complete set of nine measurements made with the specimen in the new position.

Report

5. The individual observations shall be recorded to the nearest 0.01 in., and the average of the nine measurements expressed to the nearest 0.05 in. shall be reported as the length of the concrete core.

Standard Method of Test for

FLEXURAL STRENGTH OF CONCRETE (USING SIMPLE BEAM WITH THIRD-POINT LOADING)[1]

A.S.T.M. Designation: C 78 – 44

ADOPTED, 1938; REVISED, 1939, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 78; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test covers the procedure for determining the flexural strength of concrete by the use of a simple beam with third-point loading.

NOTE.—For methods of molding concrete specimens see the Tentative Method of Making and Curing Concrete Compression and Flexure Test Specimens in the Laboratory (A.S.T.M. Designation: C 192),[3] and the Standard Method of Making and Curing Concrete Compression and Flexure Test Specimens in the Field (A.S.T.M. Designation: C 31).[3]

Apparatus

2. The third-point loading method shall be used in making flexure tests of concrete employing bearing blocks which will insure that forces applied to the beam will be vertical only and applied without eccentricity. A diagram of an apparatus which accomplishes this purpose is shown in Fig. 1.

NOTE.—Sometimes nonstandard methods of load application are used in the field. If such methods are used, the results should be correlated with those obtained with standard methods. Apparatus for making flexure tests of concrete should be designed to incorporate the following principles:

(*1*) The distance between supports and points of load application should remain constant for a given apparatus.

(*2*) The load should be applied normal to the loaded surface of the beam and in such a manner as to avoid eccentricity of loading.

(*3*) The direction of the reactions should be parallel to the direction of the applied load at all times during the test.

(*4*) The load should be applied at a uniform rate and in such a manner as to avoid shock.

(*5*) The ratio of distance between point of load application and nearest reaction to the depth of the beam should be not less than one.

The directions of loads and reactions may be maintained parallel by judicious use of linkages, rocker bearings, and flexure plates. Eccentricity of loading can be avoided by use of spherical bearings.

Test Specimen

3. The test specimen shall have a span as nearly as practicable three times its depth as tested.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates.

[2] Prior to adoption as standard, this method was published as tentative from 1930 to 1938, being revised in 1936.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Procedure

4. The test specimen shall be turned on its side with respect to its position as molded and centered on the bearing blocks. The load-applying blocks shall be brought in contact with the upper surface at the third points between the supports. If full contact is not obtained between the specimen and the load-applying blocks and the supports, due to the surfaces of the specimen being out of plane, the surfaces of the specimen, where they are in contact with the blocks or supports, shall be ground or capped (Note) to produce substantially full contact. The load may be applied rapidly up to approximately 50 per cent of the breaking load, after which it shall be applied at such a rate that the increase in extreme fiber stress does not exceed 150 psi. per min.

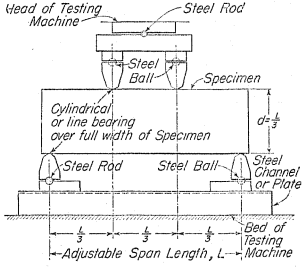


FIG. 1.—Diagrammatic View of Apparatus for Flexure Test of Concrete by Third-Point Loading Method.

NOTE.—Neat portland or alumina cement and suitable mixtures of sulfur with granular materials are recognized as suitable for capping hardened concrete specimens. Sulfur caps should be allowed to harden for at least 1 hr. before applying load, cement caps for a sufficient period to insure their resistance to cracking or flowing under the applied load.

Measurement of Specimens After Test

5. Measurements to the nearest 0.1 in. shall be made to determine the average width and average depth of the specimen at the section of failure.

Calculations

6. (*a*) If the fracture occurs within the middle third of the span length, the modulus of rupture shall be calculated as follows:

$$R = \frac{Pl}{bd^2}$$

where:

R = modulus of rupture in pounds per square inch,
P = maximum applied load indicated by the testing machine in pounds,
l = span length in inches,
b = average width of specimen in inches, and
d = average depth of specimen in inches.

NOTE.—Weight of the beam is not included in the above calculation.

(*b*) If the fracture occurs outside of the middle third of the span length by not more than 5 per cent of the span length, the modulus of rupture shall be calculated as follows:

$$R = \frac{3Pa}{bd^2}$$

where:

a = distance between line of fracture and the nearest support measured along the center line of the bottom surface of the beam, in inches.

(*c*) If the fracture occurs outside of the middle third of the span length by more than 5 per cent of the span length, the results of the test shall be discarded.

Report

7. The report shall include the following:

(*l*) Identification number,

(*2*) Average width to the nearest 0.1 in.,
(*3*) Average depth to the nearest 0.1 in.,
(*4*) Span length in inches,
(*5*) Maximum applied load in pounds,
(*6*) Modulus of rupture calculated to the nearest 5 psi.,
(*7*) Defects in specimen, and,
(*8*) Age of specimen.

Standard Method of Test for

VOLUME CHANGE OF CEMENT MORTAR AND CONCRETE[1]

A.S.T.M. Designation: C 157 - 43

ADOPTED, 1943.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 157; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. (*a*) This method of test is intended to provide a standardized procedure for determining the volume changes of cement mortar and concrete when a fairly simple, routine test is desired. It is not offered as a preferred research method or with the idea of limiting research on volume changes to a single restricted procedure. While this method is intended primarily for mixtures made with portland cement, it may also be used for testing those made with other types of cement.

(*b*) The term "volume change" is intended to apply to changes in dimension of the test specimen due to causes other than stress. The method is designed to measure volume changes due to chemical change, moisture change or similar causes, and it is intended that volume changes resulting from variations in temperature will be kept negligible. Volume change as measured and expressed in this method is reported as an increase or decrease in a *linear* dimension of the test specimen.

Apparatus

2. (*a*) *Molds for Mortar and Concrete Specimens.*—Molds for casting the prismatic test specimens shall be made of metal and so designed that the specimen will be cast with its longitudinal axis in a horizontal position. The molds shall be fitted with metal end plates that provide means for holding a stainless steel gage point (Note 1) in exact position at the center of each end of the specimen during the placing and hardening of the cement mortar or concrete. Details of a suitable mold (designed for mortar specimens) are shown in Fig. 1.

NOTE 1.—The device for holding the contact points shall be arranged so that it can be partially released after the mixture has been compacted into place in order to avoid restraint of the contact points during initial shrinkage of the specimen.

(*b*) *Measuring Apparatus.*—The apparatus for measuring volume change

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates.

[2] Prior to adoption as standard, this method was published as tentative from 1940 to 1943, being revised in 1943.

shall be of such design as to provide the following (Note 2):

(*1*) A positive means of contact with the specimen that will insure reproducible measurements of length,

(*2*) A high grade dial micrometer or other measuring device graduated to read in 0.0001-in. units, and accurate within 0.0001 in. in any 0.0010-in. range and 0.0002 in. in any 0.0100-in. range (Note 3),

(*3*) Sufficient range in the measuring device to allow for small variations in gage length of various specimens,

NOTE 3.—It is recommended that the micrometer or other measuring device be calibrated throughout its range to determine both the periodic and cumulative errors, so that proper corrections are applied to the observed data.

Test Specimens

3. (*a*) *Mortar Specimens.*—The test specimen for cement mortar shall be a prism 2 in. square and 11 in. in length.

(*b*) *Concrete Specimens.*—The test specimen for concrete shall be a prism 3 in. square and 11 in. in length.

(*c*) *Contact or Gage Points.*—Each test specimen shall have embedded at each end of the longitudinal axis, a stainless steel contact or gage point so designed as to provide an effective gage length of 10 ± 0.01 in.

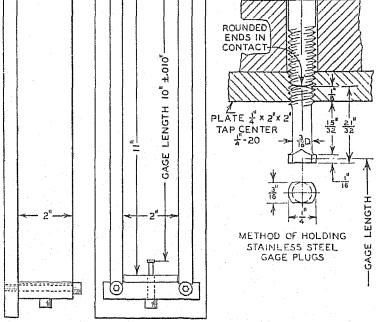


FIG. 1.—Mold for Horizontal Casting of Prismatic Cement Mortar Specimens.

(*4*) Means for checking the measuring device against a standard of reference at regular intervals, and

(*5*) Convenient and rapid measurement of specimens.

NOTE 2.—One type of apparatus that has satisfactorily met in service the requirements prescribed is shown in Fig. 2. In this apparatus the contact surfaces are provided by spherical ground ends on the contact points in the specimen and conical recesses in the terminals of the instrument.

Molding Specimens

4. (*a*) Prior to molding specimens, the inside surfaces of the molds shall be coated with a thin film of a mineral oil, petroleum jelly, or stearic acid paste.

Joints in molds may be sealed with hot paraffin or a mixture of paraffin and rosin.

(*b*) All materials shall be proportioned by weight. The mixing and molding procedure shall follow, as nearly as possible, those prescribed in the Standard Method of Test for Flexural Strength of Concrete (Using Simple Beam with Third-Point Loading) (A.S.T.M. Designation: C 78).[3]

Storage of Specimens

5. (*a*) Immediately after molding, the test specimens in the molds shall be covered with wet burlap or placed in the moist room. After 20 to 24 hr. (Note) the specimens shall be removed from the molds and placed in the storage room.

NOTE.—In the case of certain slow-hardening cements, it may be necessary to allow specimens to remain in the mold for more than 24 hr. in order to avoid damage during removal from the molds. When making tests in which such cements are included, the storage schedule may be modified but any specimens involved in the test program, which are to be used for comparison, shall be subjected to identical conditions of storage.

(*b*) The storage room shall be maintained at a temperature of 70 ± 2 F. and a relative humidity of 50 ± 5 per cent.

Procedure

6. The measuring apparatus shall be kept in the room in which the specimens are stored, and all measurements shall be made in the storage room, in order to avoid changes in temperature and humidity conditions surrounding the test specimens during length measurements.

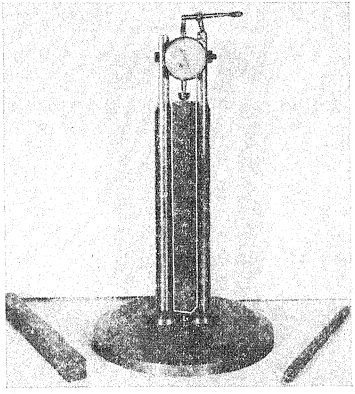


FIG. 2.—Type of Suitable Apparatus for Measurement of Volume Changes.

Report

7. The report shall include the following:

(*1*) Kind and proportions of materials used,

(*2*) Method of mixing the cement mortar or concrete,

(*3*) Method of molding specimens,

(*4*) Length of the period of preliminary moist curing and other storage conditions,

(5) Temperature and relative humidity of the storage room,

(6) Length of the period of observation, and

(7) Volume change data, recorded as expansion or contraction in inches per inch of the gage length of the test specimen.

AMERICAN ASSOCIATION STATE
HIGHWAY OFFICIALS STANDARD
A.A.S.H.O. NO.: T 121

Standard Method of Test for

WEIGHT PER CUBIC FOOT, YIELD, AND AIR CONTENT (GRAVIMETRIC) OF CONCRETE[1]

A.S.T.M. Designation: C 138 - 44

ADOPTED, 1939; REVISED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 138; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test covers the procedure for determining the weight per cubic foot of freshly mixed concrete and gives formulas for calculating: the volume of concrete produced from a mixture of known quantities of the component materials; the yield, that is, the volume of concrete per unit volume of cement; the actual cement factor; and the air content of the concrete.

NOTE.—This method of calculating air content is of value, particularly, for concrete made with air-entraining cement.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Balance.*—A balance or scale sensitive to 0.1 lb.

(*b*) *Tamping Rod.*—A straight metal rod, $\frac{5}{8}$ in. in diameter and approximately 24 in. in length with one end tapered for a distance of 1 in. to a blunt, bullet-shaped end.

(*c*) *Measure.*—A cylindrical metal measure, preferably provided with handles. The measure shall be watertight, preferably machined to accurate dimensions on the inside. It shall be reinforced around the top with a No. 10 to No. 12 U. S. gage steel band $1\frac{1}{2}$ in. in width. Depending upon the maximum nominal size of the coarse aggregate in the concrete, the measures required shall

TABLE I.—DIMENSIONAL REQUIREMENTS FOR CYLINDRICAL MEASURES.

Capacity, cu. ft.	Inside Diameter, in.	Inside Height, in.	Thickness of Metal, U. S. Gage	Maximum Nominal Size of Coarse Aggregate
½.....	10.00	11.00	No. 10 to No. 12	Up to 2 in., incl.
1.....	14.00	11.23	No. 10 to No. 12	Over 2 in.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates.

[2] Prior to adoption as standard, this method was published as tentative from 1938 to 1939.

have capacities of ½ or 1 cu. ft. and shall conform to the requirements prescribed in Table I.

Calibration of Measure

3. The measure shall be calibrated by accurately determining the weight of water at 62 F. (16.7 C.) required to fill it (Note). The factor for any measure shall be obtained by dividing the unit weight of water at 62 F. (16.7 C.), namely, 62.4 lb. per cu. ft., by the weight (in pounds) of water at 62 F. (16.7 C.) required to fill the measure.

NOTE.—Accurate filling of the measure may be secured by the use of a glass cover plate.

Sample

4. The sample of freshly-mixed concrete shall be obtained in accordance with the Standard Method of Sampling Fresh Concrete (A.S.T.M. Designation: C 172).[3]

Procedure

5. (*a*) *Sequence of Operations.*—The measure shall be filled to one third of its capacity and the mass of concrete rodded with the number of strokes prescribed in Paragraph (*b*), evenly distributed over the cross-section. The measure shall then be tapped, and filled to two thirds of its capacity, the mass of concrete again rodded, the measure tapped as before, and finally filled to overflowing, rodded and tapped as before.

(*b*) *Rodding.*—In rodding the first layer, the rod shall not strike forcibly the bottom of the measure. In rodding the second and final layers, only enough force shall be used to cause the rod to penetrate the surface of the previous layer. When the ½-cu. ft. measure is used, each layer shall be rodded with 25 strokes, and when the 1-cu. ft. measure is used each layer shall be rodded with 50 strokes.

(*c*) *Tapping.*—The exterior surface of the measure shall be tapped smartly 10 to 15 times or until no large bubbles of air appear on the surface of the rodded layer.

(*d*) *Strike-off, Cleaning, and Weighing.*—After consolidation of the concrete, the top surface shall be struck off and finished smoothly with a flat cover plate using great care to leave the measure just level full. All excess concrete shall then be cleaned from the exterior and the filled measure weighed to the nearest 0.1 lb.

Calculations

6. (*a*) *Weight per Cubic Foot.*—The net weight of the concrete shall be calculated by subtracting the weight of the measure from the gross weight. The weight per cubic foot shall be calculated by multiplying the net weight by the factor for the measure used, determined as described in Section 3.

(*b*) *Volume of Concrete.*—The volume of concrete produced per batch shall be calculated as follows:

$$S = \frac{(N \times 94) + W_f + W_c + W_w}{W}$$

where:

S = volume of concrete produced per batch, in cubic feet,
N = number of bags of cement in the batch,
94 = net weight of a bag of cement, in pounds,
W_f = total weight of fine aggregate in batch in condition used, in pounds,
W_c = total weight of coarse aggregate in batch in condition used, in pounds,
W_w = total weight of mixing water added to batch, in pounds, and
W = weight of concrete, in pounds per cubic foot.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(*c*) *Yield.*—The yield shall be calculated as follows:

$$Y = \frac{S}{N}$$

where:

Y = yield of concrete produced per 94 pound bag of cement, in cubic feet,
S = volume of concrete produced per batch, in cubic feet, and
N = number of bags of cement in the batch.

(*d*) *Cement Factor.*—The "actual" cement factor shall be calculated as follows:

$$N_1 = \frac{27}{Y} \quad \text{or} \quad N_1 = \frac{27N}{S}$$

N_1 = number of bags of cement per cubic yard of concrete produced (actual cement factor)
Y = yield of concrete produced per 94 pound bag of cement, in cubic feet,
N = number of bags of cement in the batch, and
S = volume of concrete produced per batch, in cubic feet.

(*e*) *Air Content.*—The air content shall be calculated as follows:

$$A = \frac{T - W}{T} \times 100$$

or by the formula

$$A = \frac{S - V}{S} \times 100$$

where:

A = air content (percentage of voids) in the concrete,
T = theoretical weight of the concrete, in pounds per cubic foot, computed on an air-free basis (Note),
W = weight of concrete, in pounds per cubic foot,
S = volume of concrete produced per batch, in cubic feet, and
V = total absolute volume of the component ingredients in the batch, in cubic feet.

NOTE.—The theoretical weight per cubic foot is, customarily, a laboratory determination, the value for which is assumed to remain constant for all batches made, using identical component ingredients and proportions. It is calculated from the formula:

$$T = \frac{W_1}{V}$$

where:

T = theoretical weight of the concrete, in pounds per cubic foot, computed on an air-free basis,
W_1 = total weight of the component ingredients in the batch, in pounds, and
V = total absolute volume of the component ingredients in the batch, in cubic feet.

The absolute volume of each ingredient is equal to the quotient of the weight of that ingredient divided by the product of its specific gravity times 62.4. For the aggregate components, the bulk specific gravity and weight should be based on the saturated, surface-dry condition. For the cement, a value of 3.15 may be used unless the actual specific gravity is determined by the Standard Method of Test for Specific Gravity of Hydraulic Cement (A.S.T.M. Designation: C 188).[3]

Standard Definitions of

TERMS RELATING TO CONCRETE AND CONCRETE AGGREGATES[1]

A.S.T.M. Designation: C 125 - 44

ADOPTED, 1939; REVISED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 125; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Admixture.—In the case of concrete, a material other than cement, water and aggregate, mixed or to be mixed into concrete, or to any of the ingredients thereof.

Blast-Furnace Slag.—The nonmetallic product consisting essentially of silicates and alumino-silicates of lime, and of other bases, which is developed simultaneously with iron in a blast furnace.

Crushed Gravel.—The product resulting from the artificial crushing of gravel with substantially all fragments having at least one face resulting from fracture.

Crushed Stone.—The product resulting from the artificial crushing of rocks, boulders or large cobblestones, substantially all faces of which have resulted from the crushing operation.

Elongated Piece.—In the case of concrete aggregate, one in which the ratio of the length to width of its circumscribing rectangular prism is greater than a specified value.

Fineness Modulus.—In the case of aggregate for concrete, an empirical factor obtained by adding the total percentages of a sample of the aggregate retained on each of a specified series of sieves, and dividing the sum by 100.

NOTE.—The sieve sizes used are No. 100 (149-micron), No. 50 (297-micron), No. 30 (590-micron), No. 16 (1190-micron), No. 8 (2380-micron), and No. 4 (4760-micron) and ¾ in., 3 in., 1½ in., and larger, increasing in the ratio of 2 to 1.

Flat Piece.—In the case of concrete aggregate, one in which the ratio of the width to thickness of its circumscribing rectangular prism is greater than a specified value.

Gravel.—The coarse granular material, larger than sand, resulting from the natural erosion of rock.

Water-Cement Ratio.—The ratio of the amount of water, exclusive only of that absorbed by the aggregates, to the amount of cement in a concrete mixture. This ratio is variously stated as follows: (1) by bulk volume of cement (assuming cement to weigh 94 lb. per cu. ft.); (2) by absolute volume of cement; (3) by weight; and (4) in terms of gallons of water per 94 lb. sack of cement.

[1] Under the standardization procedure of the Society, these definitions are under the jurisdiction of the A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates.

[2] Prior to adoption as standard, these definitions were published as tentative from 1936 to 1939.

Standard Specifications for

SODIUM SILICATE FOR CURING CONCRETE[1]

A.S.T.M. Designation: C 111 – 36

ADOPTED, 1936.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 111; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover sodium silicate suitable for application to the surface of concrete for the purpose of preventing moisture loss during curing.

Properties and Tests

2. (*a*) The sodium silicate shall have a density of not less than 42.25° Baumé at 60 F. (15.5 C.).

(*b*) The ratio of sodium oxide, Na_2O, to silica, SiO_2, shall be 1 Na_2O to not less than 3.2 SiO_2.

Packing and Marking

3. The sodium silicate shall be delivered on the job in containers of suitable size and of a type which will not cause any deterioration of the product prior to its application to the concrete. The name of the manufacturer, the actual net weight, and the manufacturer's grade designation shall be plainly marked on each container.

Inspection and Sampling

4. Sodium silicate may be sampled for purpose of tests either at the plant prior to delivery or on the site of the work, at the option of the purchaser. At least one sample from each 500 gal. shall be taken to determine compliance with these specifications.

Rejection

5. The sodium silicate shall be rejected if it fails to meet any of the requirements of these specifications.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates.

[2] Prior to adoption as standard, these specifications were published as tentative from 1934 to 1936. Editorially revised and rearranged in 1939.

Standard Specifications for

CRUSHED STONE AND CRUSHED SLAG FOR BITUMINOUS MACADAM BASE AND SURFACE COURSES OF PAVEMENTS[1]

A.S.T.M. Designation: D 693 - 44

ADOPTED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 693; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover the quality and grading of crushed stone and crushed slag suitable for use in the construction of bituminous macadam base and surface courses of pavements.

Basis of Purchase

2. In adapting these specifications to any particular locality or project, the type or types of aggregate (crushed stone or crushed slag) to be supplied shall be specified.

General Characteristics

3. (*a*) *Crushed Stone.*—The crushed stone shall consist of clean, tough, durable fragments free of an excess of thin or elongated pieces, and reasonably free of soft or disintegrated pieces, stone coated with dirt, or other objectionable matter.

(*b*) *Crushed Slag.*—The crushed slag shall be air-cooled blast-furnace slag and shall consist of fragments reasonably uniform in density and quality and reasonably free of thin or elongated pieces, dirt, or other objectionable matter.

Physical Properties

4. (*a*) *Wear.*—The aggregates shall conform to the following requirements for resistance to wear:

	Aggregate for Use in Base Courses	Aggregate for Use in Surface Courses
Wear, Los Angeles abrasion machine, max., per cent	50	40

(*b*) *Unit Weight of Slag.*—Crushed slag shall conform to the following requirements for unit weight, compacted:

	Crushed Slag for Use in Base Courses	Crushed Slag for Use in Surface Courses
Weight per cubic foot, compacted, min., lb.	65	70

NOTE.—The requirements for physical properties of aggregates incorporated in these specifications are recommended as the minimum requirements for quality. It is not expected, however, that these limits for quality will be applicable to all localities and conditions.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] Prior to adoption as standard, these specifications were published as tentative from 1942 to 1944, being revised in 1944.

These specifications are in effect a revision and consolidation of, and replace the former Tentative Specifications for Crushed Stone for Bituminous Macadam Base and Surface Courses (D 192 - 41 T) and for Crushed Slag for Bituminous Macadam Base and Surface Courses (D 487 - 42 T), which specifications were accordingly discontinued in 1942.

TABLE I.—GRADING REQUIREMENTS.

Size Number	Nominal Size (Sieves With Square Openings)	Amounts Finer than Each Laboratory Sieve (Square Openings), per cent by weight												
		4 in.	3½ in.	3 in.	2½ in.	2 in.	1½ in.	1 in.	¾ in.	½ in.	⅜ in.	No. 4 (4760-micron)	No. 8 (2380-micron)	No. 16 (1190-micron)
1	3½ to 1½ in.	100	90 to 100	....	25 to 60	....	0 to 15	....	0 to 5	....	....	....	....	....
2	2½ to 1½ in.	....	....	100	90 to 100	35 to 70	0 to 15	....	0 to 5	....	....	....	....	....
3	2 to 1 in.	....	....	....	100	90 to 100	35 to 70	0 to 15	....	0 to 5	....	....	....	....
5	1 to ¾ in.	....	....	....	....	....	100	90 to 100	40 to 75	15 to 35	0 to 15	0 to 5	....	....
6	¾ to ⅜ in.	....	....	....	....	....	....	100	90 to 100	20 to 55	0 to 15	0 to 5	....	....
67	¾ in. to No. 4	....	....	....	....	....	....	100	90 to 100	....	20 to 55	0 to 10	0 to 5	....
7	½ in. to No. 4	....	....	....	....	....	....	....	100	90 to 100	40 to 70	0 to 15	0 to 5	....
78	½ in. to No. 8	....	....	....	....	....	....	....	100	90 to 100	40 to 75	5 to 25	0 to 5	....
8	⅜ in. to No. 8	....	....	....	....	....	....	....	....	100	85 to 100	10 to 30	0 to 10	....
9	No. 4 to No. 16	....	....	....	....	....	....	....	....	....	100	85 to 100	10 to 40	0 to 10

Sizes

5. The sizes of aggregates for the various uses shall conform to the following requirements (Note):

	Size Number
Penetration course	1, 2, 3
Choke	5, 6, 67, 7, 78
Seal coat	7, 78, 8, 9

NOTE.—Sizes have been selected from the Tentative Specifications for Standard Sizes of Coarse Aggregate for Highway Construction (A.S.T.M. Designation: D 448).[2] An appropriate size should be selected for each use, depending on thickness of the base or surface course, quality of the aggregate, and on other local construction and service conditions.

For the harder aggregates, any of the sizes specified for penetration course aggregate may be appropriate, depending upon characteristics of the construction. For softer aggregates which tend to break down under the roller, the larger sizes should be used. The line of demarcation between harder and softer aggregates may be fixed conveniently by selection of limiting percentages of wear, Los Angeles machine, depending on characteristics of aggregates available in each locality.

The size of choke aggregate best suited for the work depends upon the size of surface voides in the penetration course and on fluidity of the bituminous binder. The more fluid binders require a densely graded choke aggregate (sizes 67 or 78), while the open gradations of aggregate are more suitable for the more viscous hot-application binders. The following combinations of size numbers are suggested:

Size of Penetration Course Aggregate	Size of Choke Aggregate	
	Viscous Binders	Fluid Binders
1	5	67
2	5 or 6	67 or 78
3	6 or 7	78

The size of seal-coat aggregate depends upon the type of surface desired.

Sieve Analysis

6. The several sizes of aggregates, when tested by means of laboratory sieves having square openings, shall conform to the requirements shown in Table I.

Methods of Sampling and Testing

7. The aggregates shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(*a*) *Sampling.*—Tentative Methods of Sampling Stone, Slag, Gravel, Sand, and Stone Block for Use as Highway Materials (A.S.T.M. Designation: D 75).[3]

(*b*) *Wear.*—Standard Method of Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine (A.S.T.M. Designation: C 131).[3]

(*c*) *Weight per Cubic Foot.*—Standard Method of Test for Unit Weight of Aggregate (A.S.T.M. Designation: C 29).[3]

(*d*) *Sieve Analysis.*—Standard Method of Test for Sieve Analysis of Fine and Coarse Aggregates (A.S.T.M. Designation: C 136).[3]

[2] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for

CRUSHED STONE, CRUSHED SLAG, AND GRAVEL FOR WATER-BOUND MACADAM BASE AND SURFACE COURSES OF PAVEMENTS[1]

A.S.T.M. Designation: D 694 - 44

ADOPTED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 694; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover the quality and grading of crushed stone, crushed slag, and gravel suitable for use in the construction of water-bound macadam base and surface courses of pavements.

Basis of Purchase

2. In adapting these specifications to any particular locality or project, the type or types of aggregate (crushed stone, crushed slag, or gravel) to be supplied shall be specified.

General Characteristics

3. (*a*) *Crushed Stone.*—The crushed stone shall consist of clean, tough, durable fragments free of an excess of thin or elongated pieces, and reasonably free of soft or disintegrated pieces, dirt, or other objectionable matter.

(*b*) *Crushed Slag.*—The crushed slag shall be air-cooled blast-furnace slag and shall consist of fragments reasonably uniform in density and quality and reasonably free of thin or elongated pieces, dirt, or other objectionable matter.

(*c*) *Gravel.*—The gravel shall consist of clean, tough, durable pieces free of an excess of thin or elongated pieces, and reasonably free of soft or disintegrated pieces, dirt, or other objectionable matter. The gravel shall be crushed and uncrushed, as specified in Section 5(*b*). Crushed gravel shall consist of fragments having two or more fractured surfaces.

(*d*) *Screenings.*—The screenings shall have a suitable binding quality.

Physical Properties

4. (*a*) *Wear.*—The aggregates shall conform to the following requirements for resistance to wear:

	Aggregate for Use in Base Courses	Aggregate for Use in Surface Courses
Wear, Los Angeles abrasion machine, max., per cent.....	60	40

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] Prior to adoption as standard, these specifications were published as tentative from 1942 to 1944.

These specifications are in effect a revision and consolidation of, and replace the former Tentative Specifications for Crushed Slag for Water-Bound Base and Surface Courses (D 488 - 38 T) and for Crushed Stone for Water-Bound Base and Surface Courses (D 489 - 40 T), which specifications were accordingly discontinued in 1942.

(*b*) *Unit Weight of Slag.*—Crushed slag shall conform to the following requirements for unit weight, compacted:

	Crushed Slag for Use in Base Courses	Crushed Slag for Use in Surface Courses
Weight per cubic foot, compacted, min., lb...	65	70

NOTE.—The requirements for physical properties of aggregates incorporated in these specifications are recommended as the minimum requirements for quality. It is not expected, however, that these limits for quality will be applicable to all localities and conditions.

(*b*) *Crushed Pieces in Gravel.*—Gravel shall contain not less than 75 per cent by weight of crushed pieces.

Sieve Analysis

6. The several sizes of aggregates, when tested by means of laboratory sieves having square openings, shall conform to the requirements shown in Table I.

Methods of Sampling and Testing

7. The aggregates shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(*a*) *Sampling.*—Tentative Methods of Sampling Stone, Slag, Gravel, Sand, and Stone Block for Use as Highway Materials (A.S.T.M. Designation: D 75).[3]

(*b*) *Wear.*—Standard Method of Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine (A.S.T.M. Designation: C 131).[3]

(*c*) *Weight per Cubic Foot.*—Standard Method of Test for Unit Weight of Aggregate (A.S.T.M. Designation: C 29).[3]

(*d*) *Sieve Analysis.*—Standard Method of Test for Sieve Analysis of Fine and Coarse Aggregates (A.S.T.M. Designation: C 136).[3]

TABLE I.—GRADING REQUIREMENTS.

Size Number	Nominal Size (Sieves with Square Openings)	Amounts Finer than Each Laboratory Sieve (Square Openings), per cent by weight											
		4 in.	3½ in.	3 in.	2½ in.	2 in.	1½ in.	1 in.	¾ in.	½ in.	⅜ in.	No. 4 (4760-micron)	No. 100 (149-micron)
1	3½ to 1½ in.....	100	90 to 100	...	25 to 60	.....	0 to 15	...	0 to 5	...	...	...	...
2	2½ to 1½ in.....	...	...	100	90 to 100	35 to 70	0 to 15	...	0 to 5	...	...	...	...
3	2 to 1 in.....	...	...	...	100	90 to 100	35 to 70	0 to 15	...	0 to 5	...	...	...
10	No. 4 to 0.....	...	...	...	...	...	...	...	...	...	100	85 to 100	10 to 30

Sizes

5. (*a*) The sizes of aggregates for the various uses shall conform to the following requirements (Note):

	Size Number
Coarse aggregate.....	1, 2, 3
Screenings.....	10

NOTE.—An appropriate size should be selected for each use, depending on local construction and service conditions. Sizes have been selected from the Tentative Specifications for Standard Sizes of Coarse Aggregate for Highway Construction (A.S.T.M. Designation: D 448) of the American Society for Testing Materials.[3]

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for

GRANITE BLOCK FOR PAVEMENTS[1]

A.S.T.M. Designation: D 59 – 39

ADOPTED, 1922; REVISED, 1926, 1939.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 59; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover granite block for pavements.

Material

2. The blocks shall be of granite of medium-size grain, showing an even distribution of constituent minerals. They shall be of uniform quality and texture throughout, and free from seams or disintegrated materials.

Physical Properties

3. The average of three tests on sample blocks shall conform to the following requirements as to physical properties:

FOR HEAVY TRAFFIC	
Percentage of wear, not more than	3.6
(or French coefficient of wear, not less than	11)
Toughness, not less than	9
FOR MODERATELY HEAVY TRAFFIC	
Percentage of wear, not more than	5.0
(or French coefficient of wear, not less than	8)
Toughness, not less than	7

Methods of Testing

4. The properties enumerated in Section 3 shall be determined in accordance with the following methods of the American Society for Testing Materials:

(*a*) *Percentage of Wear and French Coefficient of Wear.*—Standard Method of Test for Abrasion of Rock by Use of the Deval Machine (A.S.T.M. Designation: D 2),[3] except that the sample shall be prepared by use of a press and the fragments shall be as uniform and as nearly cubical as practicable.

(*b*) *Toughness.*—Standard Method of Test for Toughness of Rock (A.S.T.M. Designation: D 3).[3]

Dimensions

5. The blocks shall conform to the following requirements as to dimensions:

Length on top, in.	8 to 12
Width on top, in.	$3\frac{1}{2}$ to $4\frac{1}{2}$ or $4\frac{1}{2}$ to $5\frac{1}{2}$
Depth, in.	$4\frac{3}{4}$ to $5\frac{1}{4}$

Dressing

6. (*a*) The blocks shall be so dressed that the faces will be approximately rectangular in shape, and the ends and sides sufficiently smooth to permit the blocks to be laid with joints not exceeding $\frac{1}{2}$ in. in width at the top, and for

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] Prior to adoption as standard, these specifications were published as tentative from 1919 to 1922, being revised in 1922.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

1 in. downward therefrom, and not exceeding 1 in. in width at any other part of the joint.

(*b*) The wearing surface of the blocks shall show no depressions more than $\frac{3}{8}$ in. in depth, and the edges and corners shall be unchipped and unbroken.

Inspection

7. At least six blocks shall be selected for physical tests by the engineer or his authorized representative, subsequent to delivery at the place of use, so as to fairly represent actual deliveries. No sample shall include blocks that would be rejected by a visual examination. The bedding plane shall be marked on at least two of the blocks selected.

Rejection

8. All deliveries shall be subjected to further inspection at the place of use, prior to and during laying. All blocks which fail to conform to the requirements of Sections 2, 5, and 6 shall be rejected.

Standard Specifications for

RECUT GRANITE BLOCK FOR PAVEMENTS[1]

A.S.T.M. Designation: D 131 – 39

ADOPTED, 1923; REVISED, 1939.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 131; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover recut granite block for pavements.

Material

2. The blocks shall be of granite of medium-size grain, showing an even distribution of constituent minerals. They shall be of uniform quality and texture throughout, and free from seams or disintegrated materials.

Physical Properties

3. The average of three tests on sample blocks shall conform to the following requirements as to physical properties:

Percentage of wear	not more than 5
(or French coefficient of wear	not less than 8)
Toughness	not less than 7

Methods of Testing

4. The properties enumerated in Section 3 shall be determined in accordance with the following methods of the American Society for Testing Materials:

(*a*) *Percentage of Wear and French Coefficient of Wear.*—Standard Method of Test for Abrasion of Rock by Use of the Deval Machine (A.S.T.M. Designation: D 2),[3] except that the sample shall be prepared by use of a press and the fragments shall be as uniform and as nearly cubical as practicable.

(*b*) *Toughness.*—Standard Method of Test for Toughness of Rock (A.S.T.M. Designation: D 3).[3]

Dimensions

5. The blocks shall conform to the following requirements as to dimensions:

Length on top, in.	6 to 12
Width on top, in.	$3\frac{1}{2}$ to $4\frac{1}{2}$
Depth, in.	$4\frac{3}{4}$ to $5\frac{1}{4}$ [a]

[a] The depth may be varied by the engineer between 4 and 5 in., depth of blocks to suit the old material, provided that the blocks used on one contract shall vary not more than $\frac{1}{4}$ in. above or below the depth specified.

Dressing

6. (*a*) The blocks shall be so dressed that the faces will be approximately

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] Prior to adoption as standard, these specifications were published as tentative from 1922 to 1923, being revised in 1923.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

rectangular in shape, and the ends and sides sufficiently smooth to permit the blocks to be laid with joints not exceeding $\frac{1}{2}$ in. in width at the top, and for 1 in. downward therefrom, and not exceeding 1 in. in width at any other part of the joint.

(*b*) The wearing surface of the blocks shall show no depressions more than $\frac{3}{8}$ in. in depth, and the edges and corners shall be unchipped and unbroken.

Inspection

7. At least eight blocks shall be selected for physical tests by the engineer or his authorized representative, subsequent to delivery at the place of use, so as to fairly represent actual deliveries. No sample shall include blocks that would be rejected by a visual examination. The bedding plane shall be marked on at least two of the blocks selected.

Rejection

8. All deliveries shall be subjected to further inspection at the place of use, prior to and during laying. All blocks which fail to conform to the requirements of Sections 2, 5, and 6 shall be rejected.

Standard Specifications for

GRANITE BLOCK FOR DURAX PAVEMENTS[1]

A.S.T.M. Designation: D 132 – 39

ADOPTED, 1923; REVISED, 1939.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 132; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover granite block for Durax pavements.

Material

2. The blocks shall be of granite of medium-size grain, showing an even distribution of constituent minerals. They shall be of uniform quality and texture throughout, and free from seams or disintegrated materials.

Physical Properties

3. The average of three tests on sample blocks shall conform to the following requirements as to physical properties:

Percentage of wear	not more than 5
(or French coefficient of wear	not less than 8)
Toughness	not less than 7

Methods of Testing

4. The properties enumerated in Section 3 shall be determined in accordance with the following methods of the American Society for Testing Materials:

(*a*) *Percentage of Wear and French Coefficient of Wear.*—Standard Method of Test for Abrasion of Rock by Use of the Deval Machine (A.S.T.M. Designation: D 2),[3] except that the sample shall be prepared by use of a press and the fragments shall be as uniform and as nearly cubical as practicable.

(*b*) *Toughness.*—Standard Method of Test for Toughness of Rock (A.S.T.M. Designation: D 3).[3]

Dimensions

5. The blocks shall be not less than 3 in. nor more than 4 in. in length, width, or depth.

Dressing

6. (*a*) The blocks shall be so dressed that the faces will be approximately rectangular in shape, and the ends and sides sufficiently smooth to permit the blocks to be laid with joints not exceeding $\frac{1}{2}$ in. in width at the top, and for 1 in. downward therefrom, and not exceeding 1 in. in width at any other part of the joint.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] Prior to adoption as standard, these specifications were published as tentative from 1922 to 1923.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(*b*) The wearing surface of the blocks shall show no depressions more than $\frac{3}{8}$ in. in depth, and the edges and corners shall be unchipped and unbroken.

Inspection

7. At least 25 blocks shall be selected for physical tests by the engineer or his authorized representative, subsequent to delivery at the place of use, so as to fairly represent actual deliveries. No sample shall include blocks that would be rejected by a visual examination. The bedding plane shall be marked on at least two of the blocks selected.

Rejection

8. All deliveries shall be subjected to further inspection at the place of use, prior to and during laying. All blocks which fail to conform to the requirements of Sections 2, 5, and 6 shall be rejected.

APPROVED AS
AMERICAN TENTATIVE STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: A31-1924

Standard Specifications for

MATERIALS FOR CEMENT GROUT FILLER FOR BRICK AND STONE BLOCK PAVEMENTS[1]

A.S.T.M. Designation: D 57 – 20

ADOPTED, 1920.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 57; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover the materials (sand, portland cement, and water) for use in cement grout filler for brick and stone block pavements.

SAND

General Characteristics

2. The sand shall consist of clean, hard, durable stone particles, preferably siliceous, free from lumps of clay and objectionable foreign matter.

Grading

3. (*a*) The sand shall be well graded from coarse to fine and when tested by means of laboratory sieves shall conform to the following requirements:

Sieve	Percentage Passing
No. 10 (2000-micron)	100
No. 20 (840-micron) not less than	80
No. 200 (74-micron) not more than	5

(*b*) The sieve analysis of the sand shall be made in accordance with Standard Method of Test for Sieve Analysis of Fine and Coarse Aggregates (A.S.T.M. Designation: C 136) of the American Society for Testing Materials.[3]

Mortar Strength

4. (*a*) The sand shall be mixed with Portland cement conforming to the requirements for type I of the Standard Specifications for Portland Cement (A.S.T.M. Designation: C 150) of the American Society for Testing Materials,[3] in the proportion of 1 part of cement to 3 parts of sand, by weight, according to the standard method of making 1:3 mortar briquets. The resulting mortar at the age of 7 and 28 days shall have a tensile strength of at least 75 per cent of that developed in the same time by mortar of the same consistency, made of 1 part of the same cement and 3 parts of Ottawa sand.

(*b*) Preliminary acceptance samples shall be subjected to mortar strength tests at both 7 days and 28 days, and

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] Prior to adoption as standard, these specifications were published as tentative from 1919 to 1920. Editorially revised and rearranged in 1939.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

acceptance based thereon. Samples tested during the progress of the work will be accepted on the basis of the 7-day test.

(*c*) The tests for mortar strength shall be made in accordance with the Standard Methods of Test for Tensile Strength of Hydraulic-Cement Mortars (A.S.T.M. Designation: C 190).[3]

NOTE.—The portland cement used in testing the proposed sand should preferably be that used or to be used in the work.

Inspection

5. The sand shall be subject to inspection after delivery at the place of use, prior to and during the laying of the pavement.

Sampling

6. The sand shall be sampled for testing at the source of supply, if required. Samples shall also be taken for testing from time to time, at the place of use prior to and during the laying of the pavement, whenever desired by the engineer.

PORTLAND CEMENT

Cement

7. Portland cement for use in cement grout filler shall conform to the requirements for type I of the Standard Specifications for Portland Cement (A.S.T.M. Designation: C 150) of the American Society for Testing Materials.[3]

WATER

Water

8. The water used shall be clean and free from harmful amounts of oil, acid, alkali, organic or vegetable matter.

Standard Specifications for

MATERIALS FOR SAND-CEMENT BED FOR BRICK AND BLOCK PAVEMENTS[1]

A.S.T.M. Designation: D 58 – 37

ADOPTED, 1920; REVISED, 1924, 1937.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 58; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover the materials (sand and portland cement) for use in sand-cement bed for brick, stone block, wood block, asphalt block, and other block pavements.

SAND

General Characteristics

2. The sand shall consist of clean, hard, durable stone particles, preferably siliceous, free from lumps of clay and objectionable foreign matter.

Grading

3. (*a*) The sand shall be well graded from coarse to fine and when tested by means of laboratory sieves shall conform to the following requirements:

Sieve	Percentage Passing
No. 4 (4760-micron)	100
No. 8 (2380-micron) not less than	90

(*b*) The sieve analysis of the sand shall be made in accordance with the Standard Method of Test for Sieve Analysis of Fine and Coarse Aggregates (A.S.T.M. Designation: C 136) of the American Society for Testing Materials.[3]

Mortar Strength

4. (*a*) The sand shall be passed through a No. 8 (2380-micron) sieve and that portion retained by the No. 8 sieve shall be discarded. The portion passing a No. 8 sieve shall be mixed with portland cement conforming to the requirements for type I of the Standard Specifications for Portland Cement (A.S.T.M. Designation: C 150) of the American Society for Testing Materials,[3] in the proportion of 1 part cement to 3 parts of sand, by weight, according to the standard method of making 1:3 mortar briquets. The resulting mortar at the age of 7 and 28 days shall have a tensile strength of at least 75 per cent of that developed in the same time by mortar of the same consistency, made of 1 part of the same cement and 3 parts of Ottawa sand.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] Prior to adoption as standard, these specifications were published as tentative from 1919 to 1920. Editorially revised and rearranged in 1939.

These specifications comprise a revision of the Standard Specifications for Materials for Cement Mortar Bed for Brick, Stone Block, Wood Block, Asphalt Block and Other Block Pavements (A.S.T.M. Designation: D 58 – 24), which specifications they supersede.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(*b*) Preliminary acceptance samples shall be subjected to mortar strength tests at both 7 days and 28 days, and acceptance based thereon. Samples tested during the progress of the work will be accepted on the basis of the 7-day test.

(*c*) The tests for mortar strength shall be made in accordance with the Standard Methods of Test for Tensile Strength of Hydraulic-Cement Mortars (A.S.T.M. Designation: C 190).[3]

NOTE.—The portland cement used in testing the proposed sand should preferably be that used or to be used in the work.

Inspection

5. The sand shall be subject to inspection after delivery at the place of use, prior to and during the laying of the pavement.

Sampling

6. The sand shall be sampled for testing at the source of supply, if required. Samples shall also be taken for testing from time to time, at the place of use prior to and during the laying of the pavement, whenever desired by the engineer.

PORTLAND CEMENT

Cement

7. Portland cement for use in the cement mortar bed shall conform to the requirements for type I of the Standard Specifications for Portland Cement (A.S.T.M. Designation: C 150) of the American Society for Testing Materials.[3]

Standard Specifications for

MINERAL FILLER FOR SHEET ASPHALT AND BITUMINOUS CONCRETE PAVEMENTS[1]

A.S.T.M. Designation: D 242 – 39

ADOPTED, 1939.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 242; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover mineral filler for sheet asphalt and bituminous concrete pavements.

Composition

2. The mineral filler shall consist of limestone dust, portland cement, or other mineral matter from sources agreed upon by the purchaser and the seller.

Physical Properties

3. The material shall be thoroughly dry and free from lumps consisting of aggregations of fine particles. When tested by means of laboratory sieves the mineral filler shall conform to the following requirements:

Sieve	Percentage Passing
No. 200 (74-micron)	not less than 65
No. 80 (177-micron)	not less than 95
No. 30 (590-micron)	100

Method of Testing

4. The sieve analysis of the mineral filler shall be made in accordance with the Standard Method of Test for Sieve Analysis of Mineral Filler (A.S.T.M. Designation: D 546) of the American Society for Testing Materials.[3]

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] Prior to adoption as standard, these specifications were published as tentative from 1926 to 1939.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for

SAND FOR SHEET ASPHALT AND BITUMINOUS CONCRETE PAVEMENTS[1]

A.S.T.M. Designation: D 162 - 29

ADOPTED, 1929.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 162; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover sand for use in the construction of the surface course of sheet asphalt pavements and those types of bituminous concrete pavements in which sand predominates.

Physical Properties

2. (*a*) The grains of the sand shall be clean, tough, rough-surfaced, and angular. The sand after drying shall be free from lumps or balls of clay or of clay and sand. Before being incorporated into the paving mixture, it shall conform to the following requirements as to grading:

Passing Sieve		Retained on Sieve	Per cent	
No. 200	(74-micron)		0 to 5	
No. 100	(149-micron)	No. 200	6 to 25	15 to 40
No. 80	(177-micron)	No. 100	6 to 25	
No. 50	(297-micron)	No. 80	5 to 40	30 to 60
No. 40	(420-micron)	No. 50	5 to 30	
No. 30	(590-micron)	No. 40	8 to 25	
No. 20	(840-micron)	No. 30	5 to 15	18 to 50
No. 10	(2000-micron)	No. 20	5 to 15	
No. 10	(2000-micron)		95 to 100	
No. 4	(4760-micron)		100	

(*b*) *General Characteristics.*—The general characteristics shall be determined by examination under a microscope or a magnifying glass.

Sampling

3. The sand shall be sampled in accordance with the Tentative Methods of Sampling Stone, Slag, Gravel, Sand, and Stone Block for Use as Highway Materials (A.S.T.M. Designation: D 75) of the American Society for Testing Materials.[3]

Methods of Testing

4. The sieve analysis shall be made on samples of dried sand, weighing not less than 100 g., following the procedure described in Section 5 of the Standard Method of Test for Sieve Analysis of Fine and Coarse Aggregates (A.S.T.M. Designation: C 136) of the American Society for Testing Materials.[3] Determination of percentage of particles passing a No. 200 (74-micron) sieve shall be made by dry sieving.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] Prior to adoption as standard, these specifications were published as tentative from 1923 to 1929. Editorially revised and rearranged in 1939.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for

PREFORMED EXPANSION JOINT FILLERS FOR CONCRETE[1]

NONEXTRUDING AND RESILIENT TYPES

A.S.T.M. Designation: D 544 - 41

ADOPTED, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 544; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover preformed expansion joint fillers of the following four types for use in concrete construction. The type or types desired shall be specified by the purchaser.

Type I, cork,
Type II, self-expanding cork,
Type III, sponge rubber, and
Type IV, cork rubber.

Manufacture

2. (*a*) *Cork and Self-Expanding Cork.*—These types shall consist of preformed strips which have been formed from clean granulated cork particles securely bound together by a synthetic resin of an insoluble nature. The granulated cork shall be relatively free from hard particles or dust and shall not have been exposed in the process of manufacture to a temperature exceeding 300 F. (149 C.).

(*b*) *Sponge Rubber.*—This type shall consist of preformed strips composed essentially of a durable, elastic rubber compound which may be reinforced on each side with a layer of asphalt treated felt which has been bonded to the rubber compound filler under heat and pressure.

(*c*) *Cork Rubber.*—This type shall consist of preformed strips which have been formed from clean granulated cork particles securely bound together by a durable elastic rubber compound. The granulated cork shall be relatively free from hard particles or dust and shall not have been exposed in the process of manufacture to a temperature exceeding 300 F. (149 C.).

Character of Strips

3. Preformed strips of expansion joint fillers shall be of such character as not to be deformed or broken by twisting, bending, or other ordinary handling when exposed to atmospheric conditions. Pieces of the joint filler which have been damaged shall be rejected.

Properties

4. (*a*) *Recovery.*—The test specimen shall be given three applications of a load sufficient to compress the material

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] Prior to adoption as standard, these specifications were published as tentative from 1939 to 1941.

A "tentative revision" of this standard appears in the Tentative Revisions of Standards section at the back of this book.

to 50 per cent of its thickness before test. The load shall be immediately released after each application. At the end of 1 hr. after the third application, the joint shall have recovered to at least 90 per cent of its thickness before test.

(*b*) *Compression.*—The load required to compress the test specimen to 50 per cent of its thickness before test shall not be less than 100 nor more than 750 psi.

(*c*) *Extrusion.*—The test specimen shall be compressed to 50 per cent of its thickness before test with three of the edges restrained. The amount of extrusion of the free edge shall not exceed 0.25 in.

(*d*) *Expansion.*—In the case of self-expanding cork (type II) expansion joint filler only, the test specimen after being immersed in boiling water for 1 hr. shall have a final thickness not less than 140 per cent of the thickness before test. Discoloration of the water shall not be considered an indication of failure.

(*e*) *Boiling in Hydrochloric Acid.*—In the case of cork and self-expanding cork (types I and II) expansion joint fillers only, the test specimen when boiled for 1 hr. in hydrochloric acid (sp. gr. 1.19) shall show no evidence of disintegration. Discoloration, or a small amount of swelling shall not be considered as failure.

(*f*) *Weathering Test.*—A weathering test may be conducted in accordance with the procedure specified in Section 8. Test specimens shall show no evidence of disintegration when subjected to the specified weathering test. Test specimens which have been subjected to the weathering test may be required to meet the requirements for recovery, compression, and extrusion specified in Section 4 (*a*), (*b*), and (*c*).

Dimensions and Permissible Variations

5. The preformed strips shall conform to the dimensions specified or shown on the plans. Strips of the joint filler which do not conform to the specified dimensions, within the permissible variations of plus $\frac{1}{16}$ in. in thickness, plus or minus $\frac{1}{8}$ in. in depth, and plus or minus $\frac{1}{4}$ in. in length shall be rejected.

Packing

6. Self-expanding cork shall be wrapped in waterproof paper, sealed in a manner that will prevent the entrance of moisture, and packed at the plant in sizes convenient for handling on the job.

Sampling

7. (*a*) *Size of Samples.*—Each sample shall consist of sufficient material to provide at least 5 test specimens measuring $4\frac{1}{2}$ by $4\frac{1}{2}$ in.

(*b*) *Number of Samples.*—One representative sample shall be selected from each shipment of 1000 sq. ft. or fraction thereof of each thickness ordered.

(*c*) Samples shall be packed for transportation in such a manner that there will be no danger of distortion or breakage. Samples of self-expanding cork, in addition to the above, shall be kept dry as received and wrapped for transportation in a manner that will prevent the entrance of moisture.

Methods of Testing

8. The properties enumerated in these specifications shall be determined in accordance with the Standard Methods of Testing Preformed Expansion Joint Fillers for Concrete (A.S.T.M. Designation: D 545) of the American Society for Testing Materials.[3]

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for

CALCIUM CHLORIDE[1]

A.S.T.M. Designation: D 98 – 34

ADOPTED, 1934.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 98; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover calcium chloride to be used for road purposes, acceleration of concrete, and curing of concrete.

Chemical Composition

2. The calcium chloride shall conform to the following requirements as to chemical composition:

Calcium chloride, $CaCl_2$ (anhydrous), min., per cent	77
Magnesium chloride, $MgCl_2$, max., per cent	0.5
Total alkali chlorides, max., per cent	2.0
Other impurities, max., per cent.	1.0

Grading

3. The calcium chloride shall be in the form of flakes and when tested by means of laboratory sieves[3] shall conform to the following requirements:

Passing a $\frac{3}{8}$-in. sieve	100 per cent
Retained on a $\frac{1}{4}$-in. sieve, max.	20 per cent
Passing an 840-micron (No. 20) sieve, max.	10 per cent

Packing and Marking

4. The calcium chloride shall be delivered in moistureproof bags or sacks containing approximately 100 lb. each, or in airtight steel drums weighing not more than 450 lb. each The name of the manufacturer, the lot number, the approximate net weight, and the percentage of calcium chloride guaranteed by the manufacturer shall be legibly marked on each container.

Inspection

5. Every facility shall be provided the purchaser should he elect to have his representative sample the material at the plant. If the purchaser decides to sample the material after delivery it is understood that a 3 per cent variation in content of $CaCl_2$ from the chemical composition prescribed in Section 2 shall be permissible.

Rejection

6. The calcium chloride shall be rejected if it fails to conform to any of the requirements of these specifications, and if it has become caked or sticky in the containers before opening.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] Prior to adoption as standard, these specifications were published as tentative from 1921 to 1934, being revised in 1922, 1930, 1933, and 1934. Editorially revised and rearranged in 1939.

[3] Detailed requirements for these sieves are given in the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for
SODIUM CHLORIDE[1]

A.S.T.M. Designation: D 632 – 43

ADOPTED, 1943.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 632; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover sodium chloride to be used for road purposes.

Chemical Composition

2. The sodium chloride shall conform to the following requirement as to chemical composition:

Sodium chloride (NaCl), min., per cent	98.0

Grading

3. (*a*) The sodium chloride, when used in the form of rock salt, shall conform to the following requirements as determined by laboratory sieves:[3]

Retained on a No. 4 (4760-micron) sieve, max., per cent	12
Retained on a No. 8 (2380-micron) sieve, per cent	60 to 95
Passing a No. 30 (590-micron) sieve, max., per cent	7

(*b*) Evaporated salt may be used in lieu of rock salt when it conforms to the requirement as to chemical composition prescribed in Section 2. If the evaporated salt gives indications of caking, it shall be screened, and only the portion passing the No. 4 (4760-micron) sieve shall be used.

Packing and Marking

4. The sodium chloride shall be delivered in bags or sacks, drums, or bulk carload lots. The name of the manufacturer, the lot number, the approximate net weight, and the percentage of sodium chloride guaranteed by the manufacturer shall be legibly marked on each container, or in the case of carload shipments, noted on the shipping report.

Inspection

5. Every facility shall be provided the purchaser should he elect to have his representative sample the material at the place of manufacture. If the purchaser decides to sample the material after delivery, it is understood that a 0.5 per cent variation in content of sodium chloride from the chemical composition prescribed in Section 2 shall be permissible.

Rejection

6. The sodium chloride shall be rejected if it fails to conform to any of the requirements of these specifications.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] Prior to adoption as standard these specifications were published as tentative from 1941 to 1943.

[3] Detailed requirement for these sieves are given in the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Method of Test for

ABRASION OF ROCK BY USE OF THE DEVAL MACHINE[1]

A.S.T.M. Designation: D 2 - 33

ADOPTED, 1908; REVISED, 1926, 1933.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 2; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method covers the procedure for testing rock for resistance to abrasion in the Deval testing machine.

NOTE.—This well-known method of test is similar in almost all respects to the Deval abrasion test of the French School of Roads and Bridges. It has been used since 1878, and is entirely satisfactory for the purpose for which it was designed.

Apparatus

2. (*a*) *Deval Machine.*—The Deval abrasion testing machine shall consist of one or more hollow cast-iron cylinders closed at one end and furnished with a tightly fitting iron cover at the other. The inside dimensions of the cylinders shall be 20 cm. in diameter and 34 cm. in depth. These cylinders shall be mounted on a shaft at an angle of 30 deg. with the axis of rotation of the shaft.

(*b*) *Sieve.*—The No. 12 (1680-micron) square hole sieve shall conform to the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials.[3]

Test Sample

3. At least 30 lb. of coarsely broken stone shall be available for a test. The rock to be tested shall be broken into pieces as nearly uniform in size as possible, and as nearly 50 pieces as possible shall constitute a test sample. The total weight of rock used as the test sample shall be 5 kg. weighed to the nearest 10 g. All test pieces shall be washed and thoroughly dried before weighing.

Procedure

4. The test sample shall be placed in the Deval abrasion machine and the machine rotated for 10,000 revolutions at a rate of from 30 to 33 r.p.m. At the completion of the test, the material shall be removed from the machine and sieved on a No. 12 (1680-micron) sieve. The material passing the No. 12 sieve shall be considered in determining the amount of wear which shall be expressed, either as a percentage of the 5-kg. sample used in the test, or as the French coefficient of wear calculated as follows:

$$\text{Coefficient of wear} = 20 \times \frac{20}{W} = \frac{400}{W}$$

where: W = weight in grams of the detritus under 0.168 cm. (0.066 in.) in size per kilogram of rock used.

[1] Under the standardization procedure of the Society this method is under the jurisdiction of the A.S.T.M Committee D-4 on Road and Paving Materials.

[2] Editorially revised and rearranged in 1939.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: A5-1930

AMERICAN ASSOCIATION STATE
HIGHWAY OFFICIALS STANDARD
A.A.S.H.O. NO.: T 5-35

Standard Method of Test for

TOUGHNESS OF ROCK[1]

A.S.T.M. Designation: D 3 – 18

ADOPTED, 1908; REVISED, 1918.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 3; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test covers the procedure for determining the toughness of rock.

Toughness

2. Toughness, as applied to rock, is the resistance offered to fracture under impact, expressed as the final height of blow required of a standard hammer to cause fracture of a cylindrical test specimen of given dimensions.

Apparatus

3. Any form of impact machine which will comply with the following essentials may be used in making the test:

(*a*) A cast-iron anvil weighing not less than 50 kg., firmly fixed upon a solid foundation.

(*b*) A hammer weighing 2 kg., arranged so as to fall freely between suitable guides.

(*c*) A plunger made of hardened steel and weighing 1 kg., arranged to slide freely in a vertical direction in a sleeve, the lower end of the plunger being spherical in shape with a radius of 1 cm.

(*d*) Means for raising the hammer and for dropping it upon the plunger from any specified height from 1 to not less than 75 cm., and means for determining the height of fall within approximately 1 mm.

(*e*) Means for holding the cylindrical test specimen securely on the anvil without rigid lateral support, and under the plunger in such a way that the center of its upper surface shall, throughout the test, be tangent to the spherical end of the plunger at its lowest point.

Sampling

4. Quarry samples of rock from which test specimens are to be prepared shall measure at least 6 in. on a side and at least 4 in. in thickness, and when possible shall have the plane of structural weakness of the rock plainly marked thereon. Samples should be taken from freshly quarried material, and only from pieces which show no evidences of incipient fracture due to blasting or other causes.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] Editorially revised and rearranged in 1939.

The samples should preferably be split from large pieces by the use of plugs and feathers and not by sledging. Commercial stone-block samples from which test specimens are to be prepared shall measure at least 3 in. on each edge.

Test Specimens

5. Specimens for test shall be cylinders 25 mm. in height and from 24 to 25 mm. in diameter, prepared as described in Section 6. Three specimens shall constitute a test set. The ends of the specimen shall be plane surfaces at right angles to the axis of the cylinder.

Preparation of Test Specimens

6. One set of specimens shall be drilled perpendicular and another parallel to the plane of structural weakness of the rock, if such plane is apparent. If a plane of structural weakness is not apparent, one set of specimens shall be drilled at random. Specimens shall be drilled in a manner that will not subject the material to undue stresses and which will insure the specified dimensions.[3] The ends of the cylinders may be sawed by means of a band or diamond saw,[4] or in any other way which will not induce incipient fracture, but shall not be chipped or broken off with a hammer. After sawing, the ends of the specimens shall be ground plane with water and carborundum or emery on a cast-iron lap until the cylinders are 25 mm. in height.

Procedure

7. The test shall consist of a 1-cm. fall of the hammer for the first blow, a 2-cm. fall for the second blow, and an increase of 1-cm. fall for each succeeding blow until failure of the test specimen occurs.

Report

8. The height of the blow in centimeters at failure shall be the toughness of the test specimen. The individual and the average toughness of three test specimens shall be reported when no plane of structural weakness is apparent. In cases where a plane of structural weakness is apparent, the individual and average toughness of the three specimens in each set shall be reported and identified. Any peculiar condition of a test specimen which might affect the result, such as the presence of seams, fissures, etc., shall be noted and recorded with the test result.

[3] The form of diamond drill described in *Bulletin No. 347*, U. S. Department of Agriculture, pp. 6–7, is recommended, and should prove satisfactory if the instructions are strictly followed.

[4] A satisfactory form of diamond saw is described in *Bulletin No. 347*, U. S. Department of Agriculture, pp. 7–9.

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Standard Method of Test for
SIEVE ANALYSIS OF MINERAL FILLER[1]

A.S.T.M. Designation: D 546-41

ADOPTED, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 546; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test covers the sieve analysis of mineral fillers used in road and paving materials.

Apparatus

2. (*a*) *Balance.*—The balance shall be sensitive to 0.05 g.

(*b*) *Sieves.*—The sieves shall conform to the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials,[3] and shall include the No. 200 (74-micron) sieve and such other sieves as may be required by the specifications for the mineral filler.

NOTE.—In general specifications require the use of the No. 200 (74-micron), No. 80 (177-micron), and No. 30 (590-micron) sieves.

Sample

3. The sample for sieve analysis shall be obtained, by quartering or by use of a sampler, from a representative sample selected from the material to be tested. Approximately 50 g. of dry material is required for each test.

Drying of Sample

4. The sample shall be dried to substantially constant weight at a temperature not exceeding 110 C. (230 F.).

Procedure

5. (*a*) The 50.0 ± 5.0 g. sample of the dried mineral filler shall be weighed to the nearest 0.1 g. and placed on the No. 200 (74-micron) sieve which shall be thoroughly clean and dry. The sieve, with pan and cover attached, shall be held in one hand in a slightly inclined position so that the sample will be well distributed over the sieve, at the same time gently striking the side about 150 times per minute against the palm of the other hand on the up stroke. The sieve shall be turned every 25 strokes about one-sixth of a revolution in the same direction. The operation shall be continued until not more than 0.05 g. passes through the sieve in 1 min. of continuous sieving.

(*b*) The portion of the sample retained on the sieve shall then be weighed to the

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] Prior to adoption as standard, this method was published as tentative from 1939 to 1941.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

nearest 0.1 g., and placed on the sieve with the next larger opening for the series selected for the sieve analysis. Sieving shall be continued in a similar manner, using successively each of the selected series of sieves in the order of increasing size of opening, and recording the weight of that portion of the sample retained on each sieve. The weight of material and percentage of the sample passing each of the sieves shall be calculated.

(*c*) Washers, shot, or slugs shall not be used on the sieves.

Machine Sieving

6. Mechanical sieving devices may be used but the filler shall not be rejected if it meets the requirements when tested by the hand method described in Section 5. When mechanical sieving devices are used their thoroughness of sieving shall be tested by using the hand method.

Report

7. The report shall include the following:

(*a*) Results of the sieve analysis reported as the total percentage passing each sieve, expressed to the nearest 0.5 per cent, and

(*b*) The method of sieving used.

Reproducibility of Results

8. The percentages obtained by the same operator in duplicate tests on portions of the same sample should not differ by more than one passing any one sieve. The percentages obtained by different operators in different laboratories should not differ by more than two passing any one sieve.

Standard Methods of Testing

PREFORMED EXPANSION JOINT FILLERS FOR CONCRETE[1]

NONEXTRUDING AND RESILIENT TYPES

A.S.T.M. Designation: D 545 - 41

ADOPTED, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 545; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods cover the following tests for preformed expansion joint fillers for concrete: expansion in boiling water, recovery, compression, extrusion, boiling in hydrochloric acid, and weathering. The recovery, compression, and extrusion tests shall be performed at a laboratory room temperature of 70 F. (21 C.) or above.

Preparation of Test Specimens

2. (*a*) In the case of cork, sponge rubber, and cork rubber (types I, III, and IV) expansion joint fillers only, five specimens for test shall be cut from each sample. Each test specimen shall be freshly and squarely cut using a metal plate 4 by 4 in. as a cutting template. The metal template shall be machined from $\frac{1}{4}$-in. plate to fit the extrusion mold described in Section 5 (*b*). The template shall fit the mold within minus 0.005 in. in length and minus 0.005 in. in width.

(*b*) In the case of self-expanding cork, (type II) expansion joint fillers only, subsequent to boiling in water as described in Section 3, the test specimens shall be permitted to air-dry for 24 hr., and shall then be cut to size and measured as described in Paragraph (*a*).

(*c*) The thickness of each test specimen shall be determined to the nearest 0.001 in.

Expansion in Boiling Water

3. In the case of self-expanding cork (type II) expansion joint filler only, five test specimens measuring $4\frac{1}{2}$ by $4\frac{1}{2} \pm 0.10$ in. shall be cut from each sample. The thickness of each test specimen shall be determined to the nearest 0.001 in. The specimens shall be immersed in boiling water for 1 hr., after which they shall be removed and allowed to cool to room temperature for 15 min. The final thickness of each specimen shall then be measured to the nearest 0.001 in.

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] Prior to adoption as standard, these methods were published as tentative from 1939 to 1941.

A "tentative revision" of this standard appears in the Tentative Revisions of Standards section at the back of this book.

Recovery and Compression

4. (*a*) *Test Specimens.*—One of the test specimens prepared as described in Section 2 (*a*) (or one of the expanded specimens prepared as described in Section 2 (*b*) in the case of self-expanding cork, type II) shall be used for these tests. In the case of cork, sponge rubber, and cork rubber (types I, III, and IV) expansion joint fillers, these tests shall be made on specimens of the material as received. However, if the cork filler (type I) fails to meet the requirements specified, check tests shall be made on specimens which have been immersed in water for 24 hr. and subsequently air dried for 24 hr. Acceptance shall be based on the results of these check tests.

(*b*) *Mounting.*—The test specimen shall be placed on a flat metal plate, and a $4\frac{1}{2}$ by $4\frac{1}{2}$ by $\frac{1}{2}$-in. metal plate ground to have plane parallel faces shall be centered on the top surface of the specimen. A simple U-shaped bridge shall be employed to support a dial or other suitable measuring device reading to 0.001 in. above the center of the specimen. A metal cylinder or other device for transferring the load from the moving head of the testing machine around the measuring apparatus to the plate covering the specimen shall be placed upon the plate. A spherical bearing block shall be mounted between the upper end of the cylinder and the moving head of the testing machine.

(*c*) *Measurement of Thickness.*—When the specimen has been mounted as described in Paragraph (*b*) and is subjected only to the pressure of the dead weight of the $4\frac{1}{2}$ by $4\frac{1}{2}$ by $\frac{1}{2}$-in. metal plate, its thickness shall be determined by means of the measuring device. When the load transferring apparatus and spherical bearing block are placed on the test specimen, some compression may result. This reduction in thickness shall be considered as part of the 50 per cent reduction in thickness to be applied (Paragraph (*d*)).

(*d*) *Recovery.*—For the determination of the percentage of recovery, the test specimen shall be given three applications of a load sufficient to compress it to 50 per cent of its thickness before test. The load shall be applied without shock and at such a rate that the specimen will be compressed approximately 0.05 in. per min. After the first and second applications the load shall be released immediately, and the specimen permitted to recover 30 min. before the load is again applied. After the third application the load shall be released immediately and the specimen shall be permitted to recover 1 hr., after which the thickness shall again be measured. The load-transferring apparatus and spherical bearing block shall be removed from the test specimen during recovery periods between compressions and following the third application of load. The percentage of recovery shall be calculated as follows:

$$\text{Recovery, per cent} = \frac{t'}{t} \times 100$$

where:

t = thickness of the specimen before test, and

t' = thickness 1 hr. after completion of the third application of load.

(*e*) *Compression.*—The total maximum load in pounds required for the first application as specified in Paragraph (*d*) shall be divided by 16 and recorded as the unit pressure in pounds per square inch.

Extrusion

5. (*a*) *Test Specimens.*—One of the test specimens prepared as described in Section 2 (*a*) (or one of the expanded

specimens prepared as described in Section 2 (*b*) in the case of self-expanding cork, type II) shall be used for this test. In the case of cork, sponge rubber, and cork rubber (types I, III, and IV) expansion joint fillers, these tests shall be made on specimens of the material as received. However, if the cork filler (type I) fails to meet the requirements of the specifications, check tests shall be made on specimens which have been immersed in water for 24 hr. and subsequently air dried for 24 hr. Acceptance shall be based on the results of these check tests.

(*b*) *Mounting.*—The test specimen shall be placed in a suitable steel mold so constructed as to confine the lateral movement of the specimen under compression to one side only. Interior dimensions of the mold shall be 4 by 4 in. with permissible variations in length and in width of plus or minus 0.015 in. Mold sides shall be of such height as to extend at least ½ in. above the test specimen. The specimen shall then be covered with a ½-in. metal plate ground to have plane parallel faces. The metal plate shall be machined to fit within the three restraining sides of the steel mold snugly but without binding. A simple U-shaped bridge shall be employed to support above the center of the specimen a dial or other suitable measuring device reading to 0.001 in. A metal cylinder or other device for transferring the load from the moving head of the testing machine around the measuring apparatus to the plate covering the specimen shall be placed upon the plate. A spherical bearing block shall be mounted between the upper end of the cylinder and the moving head of the testing machine.

(*c*) *Measurement of Thickness.*—When the specimen has been mounted as described in Paragraph (*b*) and is subjected only to the pressure of the dead weight of the 4 by 4 by ½-in. metal plate, its thickness shall be determined by means of the measuring device. When the load transferring apparatus and spherical bearing block are placed on the test specimen, some compression may result. This reduction in thickness shall be considered as part of the 50 per cent reduction in thickness to be applied (Paragraph (*d*)).

(*d*) *Extrusion.*—For the determination of the amount of extrusion, the specimen shall be given one application of a load sufficient to compress it to 50 per cent of its thickness before test. The load shall be applied without shock at such a rate that the specimen will be compressed approximately 0.05 in. per min. The amount of extrusion in inches shall be determined by measuring the maximum movement of the free edge of the test specimen during the 50 per cent compression of the specimen. The extrusion shall be measured by means of a dial or other suitable device reading to 0.001 in.

Boiling in Hydrochloric Acid

6. In the case of cork and self-expanding cork (types I and II) expansion joint fillers only, one of the test specimens prepared as described in Section 2 (*a*) (or one of the expanded specimens prepared as described in Section 2 (*b*) in the case of self-expanding cork, type II) shall be used for this test. The specimen shall be boiled for 1 hr. in hydrochloric acid (sp. gr. 1.19). The test specimen shall be examined for evidences of disintegration.

Accelerated Weathering Tests

7. (*a*) Two specimens, prepared as described in Section 2 (*a*) (or two of the of the expanded specimens prepared as described in Section 2 (*b*) in the case of self-expanding cork, type II) shall be exposed to a temperature of 165 F.

(74 C.) for a period of seven days. Upon completion of this accelerated aging test, the specimens shall be immersed in water at room temperature for 24 hr.

(*b*) The specimens shall then be placed on edge in a suitable container and water poured into the container to a depth of 2 in. (one-half the height of the specimens). It will be necessary to put a weight or simple frame across the exposed edges of the specimens during this test in order that the positions of the specimens in the water will be maintained. The pan containing the specimens partially immersed in water shall be placed in a freezing chamber for a period long enough to freeze the water into solid ice. The temperature of the freezing chamber shall be maintained between + 14 and − 4 F. (− 10 and − 20 C.). Upon completion of the freezing cycle, the pan containing the specimens shall be removed from the freezing chamber and partially immersed in water at a temperature maintained between 65 and 100 F. (18 and 38 C.). The first cycle is completed when the ice surrounding the specimens has melted entirely. This cycle shall be repeated ten times.

(*c*) The length of time required for freezing and thawing will be governed by the temperature of the freezing chamber and the volume of water around the specimens.

(*d*) After ten freezing-and-thawing cycles have been completed, the specimens shall be removed from the water and allowed to stand in air at room temperature for 48 hr. The test specimens shall be examined for evidences of disintegration.

Standard Methods of

CHEMICAL ANALYSIS OF CALCIUM CHLORIDE[1]

A.S.T.M. Designation: D 345 – 34

ADOPTED, 1934.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 345; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods cover procedures for the chemical analysis of calcium chloride.

Total Calcium

2. (*a*) Weigh accurately a sample of approximately 5 g. (Note 1) and dissolve in water. Add sufficient HCl to clear, and then make up to a volume of 500 ml. Pipette a 25.00 ml. aliquot and add enough diluted NH_4OH to make slightly alkaline. Methyl orange or red may be used as indicator. Add 20 ml. of cold saturated NH_4Cl solution and dilute to from 100 to 125 ml. Heat to boiling and, while stirring to precipitate the calcium, add 20 ml. of cold saturated $(NH_4)_2C_2O_4$. Let stand 1 min., and if the precipitate does not show a tendency to settle well, bring back to boiling and continue the stirring. Let settle 20 min. and filter through No. 42, 11-cm. Whatman filters or paper of an equivalent grade. Wash free from oxalate and chloride with hot water. Wash the precipitate back into the original beaker and dilute to about 100 ml. Dissolve the precipitate with 10 to 15 ml. of diluted H_2SO_4 (1:4), heat to about 80 C., and titrate to a pink color with 0.1 *N* $KMnO_4$. Add the paper, and finish the titration to a faint pink with the standard $KMnO_4$.

(*b*) *Calculation.*—Calculate the total calcium as percentage of $CaCl_2$, as follows:

$$\text{Percentage of } CaCl_2 = \frac{\text{ml. of 0.1 } N \text{ } KMnO_4 \times 11.1}{\text{wt. of original sample in grams}}$$

The calculated percentage of $CaCl_2$ shall be corrected for $Ca(OH)_2$ and $CaCO_3$.

Total Alkalinity and Magnesium Compounds

3. (*a*) Weigh accurately a 10.00-g. sample (Note 1), place in a 400-ml. beaker, and dissolve in 25 ml. of water. Add a drop of phenolphthalein indicator, and titrate with 0.1 *N* HCl until the brilliant pink color fades or becomes a

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] Prior to adoption as standard, these methods were published as tentative from 1932 to 1934. Editorially revised and rearranged in 1939.

A "tentative revision" of this standard appears in the Tentative Revisions of Standards section at the back of this book.

dull, slowly-returning color when tested by adding another drop or two of indicator. Then add enough of the 0.1 *N* HCl in excess to give a permanent red color after the addition of a drop of methyl orange indicator. Titrate back to the neutral point with 0.1 *N* NaOH and adjust to a faint pink color with 0.1 *N* HCl. Use these solutions for the subsequent determination of magnesium compounds.

(*b*) The net volume of acid used is sometimes calculated as calcium hydroxide for convenience, as follows:

Percentage of $Ca(OH)_2$ =

ml. of 0.1 *N* HCl × 0.0037 × 10

(*c*) To the neutral solutions add 9 ml. of 1 *N* NaOH which will precipitate $Mg(OH)_2$ along with some $Ca(OH)_2$. Filter the precipitate and transfer as much as possible to the filter. Dissolve the precipitate from the paper to the original beaker with about 10 ml. of HCl (1:1). Add 30 ml. of cold saturated NH_4Cl. With a volume of about 150 ml., heat to boiling and add from 50 to 60 ml. (or an excess) of cold saturated $(NH_4)_2C_2O_4$. Heat just to boiling while stirring, and slowly make alkaline with diluted NH_4OH. Let stand 10 to 15 min. and filter through a retentive qualitative paper such as Whatman's No. 2 or 3. Save this filtrate. Return the precipitate to the original beaker, dissolve in HCl (1:1) as before, add 15 ml. of cold saturated NH_4Cl and 10 ml. of the $(NH_4)_2C_2O_4$, heat to boiling, and slowly make alkaline with diluted NH_4OH. When the precipitate is well formed and settles readily, filter as before, and combine the two perfectly clear filtrates. Concentrate to about 250 ml. If much magnesium is present, the oxalate may precipitate. Add HCl to dissolve it. At a temperature of approximately 70 C., slowly add 10 ml. of $(NH_4)_2HPO_4$ (15 per cent). Add NH_4OH (sp.gr. 0.90) slowly until the precipitate is formed, then add excess. Let stand over night, stirring vigorously several times and also just before filtering. Filter through a Gooch crucible or a No. 42 Whatman paper, or paper of equivalent quality, and wash with cold NH_4OH (1:10). Ignite to constant weight and weigh as $Mg_2P_2O_7$. If the alkalinity is appreciable and the total magnesium low, this should be calculated as $Mg(OH)_2$. Any balance of magnesium over total alkalinity would be $MgCl_2$.

(*d*) *Calculations.*—Calculate the percentage of $Mg(OH)_2$ as follows:

Percentage of $Mg(OH)_2$ =

grams of $Mg_2P_2O_7$ × 0.524 × 10

Percentage of $Mg(OH)_2$ × 34.3 =

ml. of 0.1 *N* HCl due to $Mg(OH)_2$

If the alkalinity titration with phenolphthalein was stopped and the milliliters recorded with the brilliant pink faded, the milliliters represent actual $Ca(OH)_2$ present, as follows:

Percentage of $Ca(OH)_2$ =

ml. of 0.1 *N* HCl × 0.0037 × 10

The difference (milliliters for methyl orange end point minus milliliters for phenolphthalein end point) minus milliliters due to $Mg(OH)_2$ equals the milliliters due to $CaCO_3$, as follows:

Percentage of $CaCO_3$ =

ml. of 0.1 *N* HCl due to $CaCO_3$ × 0.0050 × 10

Percentage as $CaCl_2$ =

(ml. for $Ca(OH)_2$ + ml. for $CaCO_3$) ×

0.00555 × 10

Deduct this latter percentage from total calcium as net $CaCl_2$.

NOTE.—If a large amount of $MgCl_2$ (2 or 3 per cent) is present, the calculation of $CaCO_3$ will be inaccurate.

Total Alkali Chlorides as Sodium Chloride

4. (*a*) The chlorides KCl and NaCl shall be determined together. Where it is desired to distinguish between them, the potassium may be determined by any standard method and the two reported separately. The perchlorate method may be used for this determination. For routine work the KCl and NaCl shall be reported as percentage of NaCl. The method used shall be as follows:

Dissolve an 8.000-g. sample (Note 1) in 50 ml. of hot water, add 1 g. of c.p. $Ca(OH)_2$, mix well, and boil gently for 1 min. Dilute to exactly 100 ml. in a volumetric flask. Filter through a dry paper, discarding the first 10 ml., pipette a 25.00-ml. aliquot, add 20 ml. of water; heat to boiling and add 50 ml. of hot $(NH_4)_2C_2O_4$ (5 per cent) or $(NH_4)_2CO_3$ (4 per cent)—preferably the latter. Dilute to 100 ml. and mix well. Let stand until the temperature is about that of the room and adjust the volume.

(*b*) Finally, filter through a dry retentive filter paper, discarding the first 10 ml. and refiltering if cloudy. Pipette a 25.00-ml. aliquot in a 250-ml. beaker and add 1 ml. of HCl (sp.gr. 1.19). Evaporate to dryness on a steam bath. Thoroughly ignite in a muffle or heat-treating furnace (Note 2) at 300 to 350 C. In about 30 min., organic and ammonium salts should have been driven off. Cool, dissolve in 10 ml. of water, add 3 drops of Na_2CrO_4 or K_2CrO_4 (5 per cent) solution as indicator, and titrate with 0.02 *N* $AgNO_3$ (Note 3) to a pink end point. If the ignition was correctly done, there will be but little brown or yellow coloration due to charred organic residue to interfere with seeing the end point. Run a blank on the reagents used. If this is less than 0.5 ml. neglect it, if over 0.5 ml. subtract from the titration after deducting 0.5 ml. (1.00 ml. of 0.02 *N* $AgNO_3$ is equivalent to 0.00117 g. NaCl). In the method as given, 0.5000 g. of sample is finally titrated.

(*c*) *Calculation.*—Calculate the total alkali chlorides as sodium chloride, as follows:

$$\text{Percentage of NaCl} = \frac{\text{ml. of 0.02 } N \text{ } AgNO_3 \times 0.00117 \times 1.05 \times 100}{0.5000}$$

or

$$\text{Percentage of NaCl} = \text{ml. of 0.02 } N \text{ } AgNO_3 \times 0.246$$

RAPID TECHNICAL METHOD

5. (*a*) For ordinary purposes, where the quantity of material purchased does not warrant the employment of the foregoing method of analysis, calcium chloride is deemed as satisfactory if the total chlorides present, determined by titration with 0.1 *N* $AgNO_3$ and calculated to $CaCl_2$, is in excess of 78 per cent and if not more than a slight precipitate of $Mg(OH)_2$ appears when a 10 per cent filtered solution of the material is made alkaline with NH_4OH.

(*b*) No material shall be rejected under this technical method of analysis, and in case the calcium chloride tested by this rapid procedure does not conform to the specifications, it shall be analyzed by the methods described in Sections 2 to 4, inclusive.

EXPLANATORY NOTES

NOTE 1.—Instead of weighing out separate samples as indicated in Sections 2, 3, and 4, a 25-g. sample may be diluted to a volume of 100 ml., and 1 ml. of this solution used for the procedure in Section 2, 40 ml. for the procedure in Section 3, and 32 ml. for the procedure in

Section 4. The 1 ml. should be measured with a standard calibrated pipette, and the 40 ml. and 32 ml. with a burette. These volumes shall then be further diluted to conform to the concentrations indicated under Sections 2, 3, and 4. This handling will render accurate distribution of the insoluble $Mg(OH)_2$ and $CaCO_3$ present more difficult than using direct weighed samples.

Instead of weighing accurately a 10.00-g. sample, a sample of approximately 10 g. may be used if desired, in which case the percentage of $Ca(OH)_2$ may be calculated as follows:

$$\text{Percentage of } Ca(OH)_2 = \frac{\text{ml. of 0.1 } N \text{ HCl} \times 0.0037 \times 100}{\text{wt. of sample in grams}}$$

NOTE 2.—In case a muffle or furnace with close heat control is not available, a moderate bunsen flame played over the beaker may be used to volatilize the ammonium and organic compounds. In this case it would be advisable to control the determination by means of a known mixture having NaCl present in the same magnitude.

NOTE 3.—The 0.02 N $AgNO_3$ may be conveniently made by diluting 50.00 ml. of exactly 0.1 N solution to a 250-ml. volume. There is an empirical correction to be made on the factor which amounts to the 0.02 N value multiplied by about 1.05. This can be determined exactly by running on known mixtures having the same magnitude composition as the calcium chloride to be analyzed.

Standard Specifications for

SLOW-SETTING EMULSIFIED ASPHALT[1]

FOR FINE AGGREGATE MIXES

A.S.T.M. Designation: D 631 – 43

ADOPTED, 1943.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 631; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover a slow-setting emulsified asphalt for fine aggregate mixes in which a substantial quantity of aggregate passes a $\frac{1}{8}$-in. sieve and a portion may pass a No. 200 (74-micron) sieve.

Properties and Tests

2. (*a*) Emulsified asphalt shall be homogeneous. It shall show no separation of asphalt after thorough mixing, within 30 days after delivery, provided separation has not been caused by freezing.

(*b*) Emulsified asphalt (slow-setting) for fine aggregate mixes shall conform to the following requirements:

Viscosity, Saybolt Furol, 60 ml. at 25 C. (77 F.)	not less than 20 sec., and not more than 100 sec.
Residue by evaporation at 163 C. (325 F.) or by distillation	not less than 57 per cent, and not more than 62 per cent
Sieve test	not more than 0.05 per cent
Modified miscibility with water[a]	difference of asphalt residue not more than 4.5 per cent
Cement mixing test	not more than 2.0 per cent

[a] If the sample of emulsified asphalt being tested fails to conform to this requirement for modified miscibility, the sample shall be tested for 5-day settlement and for miscibility, and if the numerical difference between the average percentages of asphaltic residue in the 5-day settlement test is less than 3, and if the standard miscibility test shows no appreciable coagulation in 2 hr., then the emulsified asphalt shall be considered as conforming to these specifications and shall be accepted.

(*c*) *Residue.*—The residue obtained from evaporation or distillation shall conform to the following requirements:

Penetration at 25 C. (77 F.), 100 g., 5 sec.	100 to 200
Soluble in carbon disulfide:	
Petroleum asphalts	not less than 97.0 per cent
Native asphalts	not less than 95 per cent
Ash	not more than 2 per cent
Ductility at 25 C. (77 F.)	not less than 60 cm.
Specific gravity at 25 C. (77 F.)	not less than 1.00

Basis of Purchase

3. Prior to the award of the order or contract, a sample may be tested by the purchaser, and if it fails to comply with the specification requirements, the bid accompanying the sample shall be rejected. Prior to any shipments being made on projects requiring 20,000 gal. or more, a sample shall be taken, under

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] Prior to adoption as standard, these specifications were published as tentative from 1941 to 1943.

the supervision of the purchaser, from a lot of not less than 20,000 gal. of the emulsified asphalt and shall be kept in a clean, airtight, sealed container at a temperature of not less than 4 C. (40 F.) until tested.

Sampling

4. At least one sample of not less than 1 gal. shall be taken from each lot or shipment of the emulsified asphalt after arrival at its destination. The samples shall be stored in clean, airtight containers at a temperature of not less than 4 C. (40 F.) until tested.

Methods of Testing

5. The properties enumerated in these specifications shall be determined in accordance with the Standard Methods of Testing Emulsified Asphalts (A.S.T.M. Designation: D 244) of the American Society for Testing Materials.[3]

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for

MEDIUM-SETTING EMULSIFIED ASPHALT[1]

FOR COARSE AGGREGATE PLANT MIXES

A.S.T.M. Designation: D 397 - 39

ADOPTED, 1939.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 397; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover a medium-consistency emulsified asphalt for plant mixes with coarse aggregate, substantially all of which is retained on a $\frac{1}{8}$-in. sieve and with practically no material passing a No. 200 (74-micron) sieve.

Properties and Tests

2. (*a*) Emulsified asphalt shall be homogeneous. It shall show no separation of asphalt after thorough mixing, within 30 days after delivery, provided separation has not been caused by freezing.

(*b*) Emulsified asphalt for coarse aggregate plant mixes shall conform to the following requirements:

Viscosity, Saybolt Furol, 60 ml. at 25 C. (77 F.)	not less than 100 sec.
Residue by distillation	not less than 60 per cent, and not more than 65 per cent
Settlement, 5 days	not more than 5
Demulsibility,[a] 50 ml. of 0.10 *N* $CaCl_2$	not more than 30 per cent
Sieve test	not more than 0.10 per cent
Miscibility with water	no appreciable coagulation in 2 hr.
Coating test	it shall not show appreciable separation when mixed with washed, dry, clean stone for 3 min. and shall coat the stone thoroughly.

[a] The demulsibility test shall be made within 30 days from date of shipment.

(*c*) *Residue.*—The residue obtained from distillation shall conform to the following requirements:

Penetration at 25 C. (77 F.), 100 g., 5 sec.	100 to 200
Soluble in carbon disulfide:	
Petroleum asphalts	not less than 97.5 per cent
Native asphalts	not less than 95 per cent
Ash	not more than 2 per cent
Ductility at 25 C. (77 F.)	not less than 40 cm.
Specific gravity at 25 C. (77 F.)	not less than 1.00

Basis of Purchase

3. Prior to the award of the order or contract, a sample may be tested by the purchaser and if it fails to comply with the specification requirements, the bid accompanying the sample shall be rejected. Prior to any shipments being made on projects requiring 20,000 gal. or more, a sample shall be taken, under the supervision of the purchaser, from

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] Prior to adoption as standard, these specifications were published as tentative from 1934 to 1939, being revised in 1936 and 1939.

a lot of not less than 20,000 gal. of the emulsified asphalt and shall be kept in a clean, airtight, sealed container at a temperature of not less than 4 C. (40 F.) until tested.

Sampling

4. At least one sample of not less than 1 gal. shall be taken from each lot or shipment of the emulsified asphalt after arrival at its destination. The samples shall be stored in clean, airtight containers at a temperature of not less than 4 C. (40 F.) until tested.

Methods of Testing

5. The properties enumerated in these specifications shall be determined in accordance with the Standard Methods of Testing Emulsified Asphalts (A.S.T.M. Designation: D 244) of the American Society for Testing Materials.[3]

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for

MEDIUM-SETTING EMULSIFIED ASPHALT[1]

FOR RETREAD AND COARSE AGGREGATE MIXES

A.S.T.M. Designation: D 398 – 39

ADOPTED, 1939.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 398; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover a low-consistency emulsified asphalt for retread mixes with coarse aggregate, substantially all of which is retained on a $\frac{1}{8}$-in. sieve and with practically no material passing a No. 200 (74-micron) sieve.

Properties and Tests

2. (*a*) Emulsified asphalt shall be homogeneous. It shall show no separation of asphalt after thorough mixing, within 30 days after delivery, provided separation has not been caused by freezing.

(*b*) Emulsified asphalt for retread and coarse aggregate mixes shall conform to the following requirements:

Viscosity, Saybolt Furol, 60 ml. at 25 C. (77 F.)	not less than 20 sec., and not more than 100 sec.
Residue by distillation	not less than 55 per cent, and not more than 60 per cent
Settlement, 5 days	not more than 5
Demulsibility,[a] 50 ml. of 0.10 *N* $CaCl_2$	not more than 30 per cent
Sieve test	not more than 0.10 per cent
Miscibility with water	no appreciable coagulation in 2 hr.
Coating test	it shall not show appreciable separation when mixed with washed, dry, clean stone for 3 min. and shall coat the stone thoroughly.

[a] The demulsibility test shall be made within 30 days from date of shipment.

(*c*) *Residue.*—The residue obtained from distillation shall conform to the following requirements:

Penetration at 25 C. (77 F.), 100 g., 5 sec.	100 to 200
Soluble in carbon disulfide:	
Petroleum asphalts	not less than 97.5 per cent
Native asphalts	not less than 93 per cent
Ash	not more than 2 per cent
Ductility at 25 C. (77 F.)	not less than 40 cm.
Specific gravity at 25 C. (77 F.)	not less than 1.00

Basis of Purchase

3. Prior to the award of the order or contract, a sample may be tested by the purchaser and if it fails to comply with the specification requirements, the bid accompanying the sample shall be rejected. Prior to any shipments being made on projects requiring 20,000 gal. or more, a sample shall be taken, under

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] Prior to adoption as standard, these specifications were published as tentative from 1934 to 1939, being revised in 1939.

the supervision of the purchaser, from a lot of not less than 20,000 gal. of the emulsified asphalt and shall be kept in a clean, airtight, sealed container at a temperature of not less than 4 C. (40 F.) until tested.

Sampling

4. At least one sample of not less than 1 gal. shall be taken from each lot or shipment of the emulsified asphalt after arrival at its destination. The samples shall be stored in clean, airtight containers at a temperature of not less than 4 C. (40 F.) until tested.

Methods of Testing

5. The properties enumerated in these specifications shall be determined in accordance with the Standard Methods of Testing Emulsified Asphalts (A.S.T.M. Designation: D 244) of the American Society for Testing Materials.[3]

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for

MEDIUM-SETTING EMULSIFIED ASPHALT[1]

HEAVY PREMIX—SUMMER GRADE

A.S.T.M. Designation: D 399 - 39

ADOPTED, 1939.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 399; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover a high-consistency heavy-premix grade of emulsified asphalt, for plant mix or patching with coarse aggregate, substantially all of which is retained on a $\frac{1}{8}$-in. sieve and with practically no material passing a No. 200 (74-micron) sieve.

Properties and Tests

2. (*a*) Emulsified asphalt shall be homogeneous. It shall show no separation of asphalt after thorough mixing, within 30 days after delivery, provided separation has not been caused by freezing.

(*b*) Emulsified asphalt (heavy premix—summer grade) shall conform to the following requirements:

Residue by distillation	not less than 65 per cent
Sieve test	not more than 0.10 per cent
Miscibility with water	no appreciable coagulation in 2 hr.
Coating test	it shall not show appreciable separation when mixed with washed, dry, clean stone for 3 min. and shall coat the stone thoroughly.

(*c*) *Residue.*—The residue obtained from distillation shall conform to the following requirements:

Penetration at 25 C. (77 F.), 100 g., 5 sec	100 to 200
Soluble in carbon disulfide:	
Petroleum asphalts	not less than 97.5 per cent
Native asphalts	not less than 95 per cent
Ash	not more than 2 per cent
Ductility at 25 C. (77 F.)	not less than 40 cm.
Specific gravity at 25 C. (77 F.)	not less than 1.00

Basis of Purchase

3. Prior to the award of the order or contract, a sample may be tested by the purchaser and if it fails to comply with the specification requirements, the bid accompanying the sample shall be rejected. Prior to any shipments being made on projects requiring 20,000 gal. or more, a sample shall be taken, under the supervision of the purchaser, from a lot of not less than 20,000 gal. of the emulsified asphalt and shall be kept in a clean, airtight, sealed container at a temperature of not less than 4 C. (40 F.) until tested.

Sampling

4. At least one sample of not less than 1 gal. shall be taken from each lot or shipment of the emulsified asphalt after

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] Prior to adoption as standard, these specifications were published as tentative from 1934 to 1939, being revised in 1936 and 1939.

arrival at its destination. The samples shall be stored in clean, airtight containers at a temperature of not less than 4 C. (40 F.) until tested.

Methods of Testing

5. The properties enumerated in these specifications shall be determined in accordance with the Standard Methods of Testing Emulsified Asphalts (A.S.T.M. Designation: D 244) of the American Society for Testing Materials.[3]

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for

QUICK-SETTING EMULSIFIED ASPHALT[1]

FOR PENETRATION AND SURFACE TREATMENT

A.S.T.M. Designation: D 401 – 40

ADOPTED, 1940.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 401; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover emulsified asphalt (quick-setting) for penetration and surface treatment.

Properties and Tests

2. (*a*) Emulsified asphalt shall be homogeneous. It shall show no separation of asphalt after thorough mixing, within 30 days after delivery, provided separation has not been caused by freezing.

(*b*) Emulsified asphalt (quick-setting) for penetration and surface treatment shall conform to the following requirements:

Viscosity, Saybolt Furol, 60 ml. at 25 C. (77 F.)	not less than 20 sec., and not more than 100 sec.
Residue by distillation	not less than 55 per cent, and not more than 60 per cent
Settlement, 5 days	not more than 3
Demulsibility,[a] 35 ml. of 0.02 *N* $CaCl_2$	not less than 60 per cent
Sieve test	not more than 0.10 per cent

[a] The demulsibility test shall be made within 30 days from date of shipment.

(*c*) *Residue.*—The residue obtained from distillation shall conform to the following requirements:

Penetration at 25 C. (77 F.), 100 g., 5 sec.	100 to 200
Soluble in carbon disulfide:	
Petroleum asphalts	not less than 97.5 per cent
Native asphalts	not less than 95 per cent
Ash	not more than 2 per cent
Ductility at 25 C. (77 F.)	not less than 40 cm.
Specific gravity at 25 C. (77 F.)	not less than 1.00

Basis of Purchase

3. Prior to the award of the order or contract, a sample may be tested by the purchaser and if it fails to comply with the specification requirements, the bid accompanying the sample shall be rejected. Prior to any shipments being made on projects requiring 20,000 gal. or more, a sample shall be taken, under the supervision of the purchaser, from a lot of not less than 20,000, gal. of the emulsified asphalt and shall be kept in a clean, airtight, sealed, glass or black iron container at a temperature of not less than 4 C. (40 F.) until tested.

Sampling

4. At least one sample of not less than 1 gal. shall be taken from each lot or shipment of the emulsified asphalt after arrival at its destination. The samples

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

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[2] Prior to adoption as standard, these specifications were published as tentative from 1936 to 1940, being revised in 1936.

shall be stored in clean, airtight, glass or black iron containers at a temperature of not less than 4 C. (40 F.) until tested.

Methods of Testing

5. The properties enumerated in these specifications shall be determined in accordance with the Standard Methods of Testing Emulsified Asphalts (A.S.T.M. Designation: D 244) of the American Society for Testing Materials.[3]

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for

ASPHALT FILLER FOR BRICK PAVEMENTS[1]

A.S.T.M. Designation: D 241 – 43

ADOPTED, 1943.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 241; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover asphalt filler for brick pavements.

General Requirements

2. The asphalt filler shall be free from water and shall not foam when heated to a temperature of 177 C. (350 F.) for grade A, or 230 C. (446 F.) for grade B. Grade B is the low-exuding type.

Properties

3. The filler shall conform to the requirements prescribed in Table I.

Methods of Testing

4. The material shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials:

TABLE I.—REQUIREMENTS FOR ASPHALT FILLER.

	Grade A		Grade B	
	Min.	Max.	Min.	Max.
Softening point (ring-and-ball method)	167 F. (75 C.)	185 F. (85 C.)	214 F. (101 C.)	230 F. (110 C.)
Flash point (Cleveland open cup)	469 F. (243 C.)	....	482 F. (250 C.)	....
Penetration:				
0 C. (32 F.), 200 g., 1 min	15	....	12	....
25 C. (77 F.), 100 g., 5 sec	30	40	23	32
46 C. (115 F.), 50 g., 5 sec	....	80	....	47
Ductility at 25 C. (77 F.), (5 cm. per min.), cm.	3	....	1.5	....
Loss on heating at 163 C. (325 F.), 50 g., 5 hr., per cent	....	1.0	....	0.3
Total bitumen soluble in carbon disulfide, per cent	99.0	....	99.3	....
Proportion of bitumen soluble in carbon tetrachloride, per cent	99.0	....	99.3	....

(*a*) *Sampling.*—Tentative Methods of Sampling Bituminous Materials (A.S.T.M. Designation: D 140).[3]

(*b*) *Softening Point.*—Standard Method of Test for Softening Point of Bituminous Materials (Ring-and-Ball Method) (A.S.T.M. Designation: D 36).[3]

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] Prior to adoption as standard, these specifications were published as tentative from 1926 to 1943, being revised in 1939.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(*c*) *Flash Point.*—Standard Method of Test for Flash and Fire Points by Means of Open Cup (A.S.T.M. Designation: D 92).[3]

(*d*) *Penetration.*—Standard Method of Test for Penetration of Bituminous Materials (A.S.T.M. Designation: D 5).[3]

(*e*) *Loss on Heating.*—Tentative Method of Test for Loss on Heating of Oil and Asphaltic Compounds (A.S.T.M. Designation: D 6).[3]

(*f*) *Ductility.*—Standard Method of Test for Ductility of Bituminous Materials (A.S.T.M. Designation: D 113).[3]

(*g*) *Bitumen Soluble in Carbon Disulfide.*—Standard Method of Test for Determination of Bitumen (A.S.T.M. Designation: D 4).[3]

(*h*) *Bitumen Soluble in Carbon Tetrachloride.*—Standard Method of Test for Proportion of Bitumen Soluble in Carbon Tetrachloride (A.S.T.M. Designation: D 165).[3]

Standard Specifications for
COAL-TAR PITCH FOR STONE BLOCK FILLER[1]

A.S.T.M. Designation: D 112 – 30

ADOPTED, 1930.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 112; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover coal-tar pitch suitable for use as a filler between stone block in pavements.

Properties

2. The coal-tar pitch shall conform to the following requirements:

Water	0.00 per cent
Softening point (cube-in-water method)	46 to 57 C. (115 to 135 F.)[a]
Distillation test:	
Total distillate by weight, 0 to 300 C. (32 to 572 F.)	14.00 per cent, max.
Residue by weight	86.00 per cent, min.
Specific gravity at 38 C. (100.4 F.) of total distillate to 300 C. (572 F.)	1.02 min.
Softening point (cube-in-water method) of residue from distillation test	75 C. (167 F.), max.
Ductility at 50 to 100 penetration,[b] at 25 C. (77 F.)	50 cm., min.
Total bitumen (soluble in carbon disulfide)	65.0 to 80.0 per cent

[a] The softening point (cube-in-water method) specified should have a range of not over 10 F. within the above limits. The range, within the limits of 46 to 57 C. (115 to 135 F.) should vary with the use of the material, for example, if used in admixture with sand, in a northern locality or a southern locality. The softening point range within the above limits, should also vary according to the character of the paving.

[b] The penetration of the pitch shall be brought within the range of 50 to 100 penetration by heating in an open vessel with frequent stirrings at a temperature of not over 350 F.

Methods of Testing

3. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials:

(*a*) *Percentage of Water.*—Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (A.S.T.M. Designation: D 95).[3]

(*b*) *Softening Point.*—Standard Method of Test for Softening Point of Tar Products (Cube-in-Water Method) (A.S.T.M. Designation: D 61).[3]

(*c*) *Distillation.*—Standard Method of Test for Distillation of Tar Products Suitable for Road Treatment (A.S.T.M. Designation: D 20).[3]

(*d*) *Ductility.*—Standard Method of Test for Ductility of Bituminous Materials (A.S.T.M. Designation: D 113).[3]

(*e*) *Total Bitumen.*—Standard Method of Test for Determination of Bitumen (A.S.T.M. Designation: D 4).[3]

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] Prior to adoption as standard, these specifications were published as tentative from 1921 to 1930, being revised in 1922, 1923, and 1927. Editorially revised and rearranged in 1939.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for

ASPHALT PLANK[1]

A.S.T.M. Designation: D 517 – 40

ADOPTED, 1940.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 517; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover asphalt plank of two types as used for bridge floors:

(*a*) Plain asphalt plank, and

(*b*) Mineral-surfaced asphalt plank.

NOTE.—Plain asphalt plank conforming to these specifications are also suitable for waterproofing protection where a solid bridge floor carries ballasted railroad track.

Manufacture

2. Asphalt plank shall be formed by the extrusion of a mixture of asphalt, fiber, and mineral aggregate under sufficient pressure to expel the entrapped air and form a dense mass.

Mineral Surfacing

3. In the case of mineral-surfaced asphalt plank, at least 15 oz. per sq. ft. of tough, hard, durable, coarse aggregate (well-graded from No. 4 (4760-micron)[3] to ¾ in. square-opening sieves) shall be embedded under pressure into one surface of the freshly-extruded hot plank to such a depth that none extends above the intended wearing surface. The top surface of the mineral surfacing shall not be covered with the matrix of the plank proper.

Workmanship

4. The asphalt plank shall be free from defects affecting its serviceability and appearance; it shall have straight edges and square corners.

Asphalt Cement

5. The asphalt cement shall have such characteristics that, when combined with the other ingredients, a plank of desired quality will result.

Fiber

6. The fibrous material shall consist of finely divided whole threads or fiber free from lumps, and when used it shall be in a flocculent condition. This fabric and fibrous material shall be free from all foreign materials such as metal, leather, straw, sawdust, cornstalks, or other deleterious materials.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] Prior to adoption as standard, these specifications were published as tentative from 1938 to 1940.

[3] Detailed requirements for this sieve are given in the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Mineral Filler

7. The mineral filler shall consist of finely crushed slate, limestone, asbestos, silica, or other aggregate which has been proven suitable for use with asphalt cement in constructing pavement wearing surfaces.

Dimensions

8. Asphalt plank shall have the dimensions specified or shown on the plans. Tolerances of plus or minus $\frac{1}{16}$ in. in thickness, plus or minus $\frac{1}{8}$ in. in width, and plus or minus $\frac{1}{4}$ in. in length will be permitted.

NOTE.—The following thicknesses are recognized as standard: $\frac{3}{4}$ in., 1 in., $1\frac{1}{4}$ in.,$1\frac{1}{2}$ in., and 2 in.

Absorption

9. The absorption of asphalt plank shall not exceed 1.0 per cent by weight.

Brittleness

10. At least 80 per cent of the specimens of each thickness of plank tested shall not show any detrimental cracking when tested in accordance with the method described in Section 16.

Indentation

11. The indentation of asphalt plank, irrespective of thickness, when subjected to the loads prescribed in Section 17, shall be within the following limits:

(*a*) *At 77 F. (25 C.), at the end of 1 min.*—Not less than 12 mils nor more than 27 mils.

(*b*) *At 77 F. (25 C.), at the end of 10 min.*—Not more than that specified in the following table, corresponding to the indentation at 1 min.:

Indentation Recorded at 1 min., mils (to the nearest 1 mil)	Maximum Indentation at 10 min., mils (to the nearest 0.5 mil)
12	21.5
13	23.0
14	24.0
15	25.5
16	27.0
17	28.5
18	30.0
19	31.0
20	32.5
21	33.5
22	35.0
23	36.5
24	37.5
25	39.0
26	40.0
27	41.5

(*c*) *At 125 F. (51.7 C.), at the end of 30 sec.*—Not less than 35 mils nor more than 70 mils.

(*d*) *At 125 F. (51.7 C.), at the end of 300* sec.—Not more than that specified in the following table (or interpolated therefrom) corresponding to the indentation at 30 sec.:

Indentation Recorded at 30 sec., mils (to the nearest 1 mil)	Maximum Indentation at 300 sec., mils (to the nearest 0.5 mil)
35	70.0
40	77.0
45	85.0
50	93.0
55	100.0
60	108.0
65	116.0
70	125.0

SAMPLING AND METHODS OF TESTING

Sampling

12. For purpose of tests, three planks shall be taken at random by the purchaser for each 1000 planks, or fraction thereof, of each thickness included in the shipment.

Inspection

13. Plank from each lot shall be examined for appearance, straightness of edges, and squareness of corners, and measured for width and length. They shall also be calipered at four scattered points each, with a micrometer having flat bearing surfaces at both contact points of not less than $\frac{1}{4}$ in. in diameter. The average of the readings shall be considered the thickness of the plank.

Weight of Mineral Surfacing

14. The weight of mineral aggregate shall be determined by removing the mineral surfacing aggregate from not less than 1 sq. ft. of mineral surfaced asphalt plank. The aggregate shall be cleaned with any suitable solvent, dried, and sieved on a No. 8 (2380-micron) sieve.[3] The amount of aggregate on the sieve shall be weighed and the weight of aggregate per square foot determined.

Absorption Test

15. A specimen 2 by 6 in. shall be cut from the asphalt plank in such a manner that all edges are freshly cut. The specimen shall be accurately weighed to the nearest 0.10 g., immersed in water

TABLE I.—NAIL SIZES REQUIRED FOR BRITTLENESS TEST.

Thickness of Asphalt Plank, in.	Nail Size	A.S.&W. Gage No.	Diameter, in.
¾	12 d.	9	0.148
1	16 d.	8	0.162
1½	20 d.	6	0.192
1¾	30 d.	5	0.207
2	30 d.	5	0.207

for 24 hr., removed, and the surface water wiped off with a slightly dampened cloth. The specimen shall then be weighed to the nearest 0.10 g. and the percentage of absorption determined. The time elapsing between the removal of the specimen from the bath and its weighing shall not exceed 1 min.

Brittleness Test

16. Two 6 by 6-in. specimens shall be cut from each of the three sample planks selected in accordance with Section 12. Five of these specimens shall be tested as follows: The specimens shall be immersed in a water bath and maintained at a temperature of 39 to 43 F. (4 to 6 C.) for not less than 3 hr., and then tested immediately upon their removal from the water bath. A smooth, common nail of the size specified in Table I shall be driven into the face at each of two diagonally opposite corners of the specimen not less than 1½ in. nor more than 2 in. from each edge, but not directly over a piece of mineral surfacing aggregate. The specimen shall be supported over its entire base with a timber block 7 by 7 by 1½ in. and the nails shall be driven vertically with repeated blows from a 10-lb. weight falling freely from a constant height of 20 in. above the level of the initial top of the nail or striking assembly. The height shall not be adjusted to compensate for the increased fall caused by the penetration of the nail. The nails shall be supported by a collar or other similar assembly that will hold the nail in a vertical position without friction. Should a nail strike a piece of aggregate covered by the planking matrix, as shown by bending of the nail or by its deflection from the vertical, the specimen shall be discarded and the test repeated on a new specimen. The nails shall penetrate through the asphalt plank, without detrimental cracking, to a depth of not less than 1 in. into the timber block. Cracking shall be considered detrimental when the cracks extend to a point nearer than ½ in. to an edge.

Indentation Test

17. Two 6 by 6-in. specimens from each set of sample planks selected in accordance with Section 12 shall be subjected to the following indentation tests: The specimens shall be brought to a temperature of 77 ± 1 F. (25 ± 0.5 C.) by immersing them in a water bath at this temperature for not less than 2 hr. nor more than 4 hr. Specimens shall also be immersed in water at this temperature during the test. A rod or needle with a hemispherical end 1 in. in diameter shall be applied to the surface (in case of mineral-surfaced

plank, the test surface of the specimen shall be the face that does not contain mineral surfacing aggregate) of the specimen with a load of 2 lb., the specimen being supported on a polished steel plate.[4] The indentation indicating micrometer dial shall be adjusted to zero and an additional load of 28 lb. applied to the rod or needle. The transfer of the 28-lb. load to the rod shall be completed within 10 sec. following the zero adjustment of the micrometer dial. The readings of the dial at the end of 1 min. and 10 min. from the time of application of the total load shall be taken as the measure of indentation. The 1-min. and 10-min. indentation readings shall be taken at three scattered points on each specimen. The average of the three sets of readings shall be recorded as the 1-min. and 10-min. indentations. The test shall be repeated at 125 ± 1 F. (51.7 ± 0.5 C.), the readings at three points being taken at the end of 30 sec. and 300 sec. If the average indentation of either specimen (or more than one single reading of the three made at each time period) measured as specified above fails to meet the specified requirements in any test, the plank represented by the sample shall be considered to have failed.

[4] The indentation test may be made with relatively simple apparatus which can be constructed with little expense in almost any laboratory. Where convenience and portability of the indentation apparatus are particularly desirable, the portable indenter devised by J. W. McBurney for indentation testing of asphalt tile (J. W. McBurney, "Indentation of Asphalt Tile," *Proceedings*, Am. Soc. Testing Mats, Vol. 34, Part II, p. 591 (1934)) complies with all the above stated requirements for apparatus to make the test. Adaptation of the McBurney indentation test method to asphalt plank has been described in a paper by H. W. Greider and Henri Marc, "Indentation and Compression Shear Tests for Determining Service Stability of Asphalt Plank," *Proceedings*, Am. Soc. Testing Mats., Vol. 37, Part II, p. 530 (1937).

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: A37.3-1943

Standard Method of Test for

DETERMINATION OF BITUMEN[1]

A.S.T.M. Designation: D 4 – 42

ADOPTED, 1927; REVISED, 1942.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 4; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test is intended for the determination of bitumen in materials containing at least 25 per cent bitumen. Bitumen may usually be expeditiously and accurately determined by method No. 1, Section 5. In the case of certain native asphalts containing finely divided mineral matter, the mineral residue is not easily retained by the filter and it is necessary to use method No. 2, Section 6, in order to obtain accurate results. Method No. 2 shall not be used unless the filter clogs unduly or unless the mineral matter passing through the filter exceeds 0.5 per cent.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Gooch Crucible*, approximately 4.4 cm. in width at the top, tapering to 3.6 cm. at the bottom, with a depth of 2.5 cm.

(*b*) *Asbestos* (amphibole), Gooch grade, acid washed, cut in pieces not exceeding 1 cm. in length, shredded, and shaken up with water.

(*c*) *Flasks.*—Two 125-ml. conical flasks such as Erlenmeyer flasks.

(*d*) *Filtering Flask.*

(*e*) *Filter Tube.*

(*f*) *Section of Rubber Tubing*, to hold the Gooch crucible on the filter tube.

(*g*) *Drying Oven.*

(*h*) *Bunsen Burner.*

(*i*) *Suction Pump.*

(*j*) *Analytical Balance.*

(*k*) *Desiccator.*

Preparation of Sample

3. The sample shall be representative, and if it contains more than 2 per cent of water it shall be dehydrated by distillation in a copper still in accordance with the Standard Method of Test for Water in Creosote (A.S.T.M. Designation: D 370) of the American Society for Testing Materials,[3] and the water-free distillate returned to the residue.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] Prior to its present adoption as standard, this method was published as standard from 1911 to 1923, but withdrawn and republished as tentative from 1923 to 1927, being revised in 1926 and 1927.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

If the material is hard and brittle, it may be ground, and dried at a temperature below the temperature of volatilization of the material.

Preparation of Gooch Crucible

4. Insert the filter tube in the stopper of the filtering flask, set the Gooch crucible in the filter tube, and connect the flask to the suction pump. Fill the crucible with some of the suspension of asbestos in water, allow it partly to settle in the crucible, and apply a light suction to draw off the water, leaving a firm mat of asbestos in the crucible. Add more suspended asbestos and repeat the process until a mat is built up that weighs 0.5 ± 0.1 g. after ignition (Note). Wash the asbestos mat thoroughly with water, dry in the oven and ignite over a bunsen burner. Cool the crucible in the desiccator, weigh, and replace it in the dry filter tube supported in the clean, dry filtering flask.

NOTE.—In the determination, the asbestos apparently adsorbs irreversibly a small amount of soluble bitumen (usually 1 to 5 mg. per gram of asbestos) which is not removed by subsequent washing with solvent. The weight of asbestos used, therefore, should be kept within the specified limits to insure reproducible results.

Method No. 1

Procedure

5. (*a*) Weigh approximately 2 g. of the sample into a tared conical flask, and add 100 ml. of c.p. carbon disulfide to the flask in small portions with continued agitation until all lumps disappear and nothing adheres to the bottom of the flask. Then stopper the flask and set aside for 15 min.

(*b*) Decant the carbon disulfide solution carefully through the asbestos mat in the prepared Gooch crucible, with or without light suction as may be necessary, retaining as much of the sediment as possible in the conical flask until the solution has drained through the mat. With a small amount of carbon disulfide wash down the sides of the flask and transfer the sediment and precipitate from the flask to the asbestos mat. The flask may be scrubbed with a feather if necessary in order to remove all of the precipitate. Wash the contents of the crucible with carbon disulfide until the washings are colorless, then apply suction to remove the carbon disulfide. Dry the crucible in the oven at 100 to 125 C. for 20 min., cool in the desiccator, and weigh. The increase in weight over the original weight is the weight of matter insoluble in carbon disulfide. Ignite the crucible at a red heat and after thorough ignition, cool, and weigh. The weight of the insoluble matter after ignition is the weight of ash.

(*c*) If insoluble matter adheres to the flask, dry the flask, and weigh. Add the increase in weight over the original weight to the weight of insoluble matter in the Gooch crucible.

(*d*) In case there is any question involving the amount of mineral matter that may have passed through the filter, evaporate the filtrate, and burn the bituminous residue. If a carbonate mineral is present in the filtrate ash, add to the ash a few drops of ammonium carbonate solution, and dry at 100 C., then heat for a few minutes to a dull red heat, and cool in the desiccator. Weigh, and add the weight of ash obtained to the weight of matter insoluble in carbon disulfide.

NOTE.—In the event that water-soluble salts which are insoluble in carbon disulfide are present, the amount of these salts may be determined in accordance with the procedure described in the 1937 Report of Committee D-4 on Road and Paving Materials.[4]

Method No. 2

Procedure

6. (*a*) Weigh approximately 2 g. of the sample into a tared conical flask,

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 37, Part I, p. 395 (1937).

and treat with 100 ml. of c.p. carbon disulfide. Stopper the flask loosely, and shake at intervals until all large particles have been broken down. Then allow the flask to remain undisturbed for 48 hr.

(*b*) Decant the solution into a similar tared flask, taking care to disturb as little of the residue as possible. Treat the residue in the first flask with 100 ml. of c.p. carbon disulfide, shake thoroughly, and allow both flasks to remain undisturbed for 48 hr.

(*c*) Carefully decant the solution from the second flask upon the prepared Gooch crucible, filtering without the use of suction. Then in a similar manner filter the solution from the first flask. Wash the filter with fresh carbon disulfide. Add carbon disulfide to the residue remaining in each flask, shake thoroughly, and allow to settle for 24 hr.

(*d*) Decant the solutions from both flasks through the filter. Again wash the residues remaining in the flasks with carbon disulfide, decanting and filtering the solutions as before. Repeat this treatment until the washings are practically colorless. Maintain the temperature between 20 and 25 C. Dry the crucible and the flasks at 100 to 125 C., and weigh. The total increase in weight over the total original weight is the weight of matter insoluble in carbon disulfide. Evaporate the filtrate containing the bitumen, burn the bituminous residue, and weigh. Add the weight of ash thus obtained to the weight of insoluble matter in the Gooch crucible and conical flasks. The sum of these weights is the total weight of matter insoluble in carbon disulfide.

Calculation and Report

7. (*a*) *Calculation.*—The weight of sample taken calculated to a water-free basis minus the total weight of matter insoluble in carbon disulfide is the weight of bitumen. The percentages of bitumen and of ash shall be calculated on the basis of water-free material.

(*b*) *Report.*—In every case the report shall indicate whether method No. 1 or No. 2 has been employed.

APPROVED AS
AMERICAN STANDARD
THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: A37.12-1943

Standard Method of Test for

PROPORTION OF BITUMEN SOLUBLE IN CARBON TETRACHLORIDE[1]

A.S.T.M. Designation: D 165 - 42

ADOPTED, 1927; REVISED, 1942.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 165; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test is intended for determining the proportion of bitumen soluble in carbon tetrachloride.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Gooch Crucible*, approximately 4.4 cm. in width at the top, tapering to 3.6 cm. at the bottom, with a depth of 2.5 cm.

(*b*) *Asbestos* (amphibole), Gooch grade, acid washed, cut in pieces not exceeding 1 cm. in length, shredded, and shaken up with water.

(*c*) *Flasks.*—Two 125-ml. conical flasks such as Erlenmeyer flasks.

(*d*) *Filtering Flask.*

(*e*) *Filter Tube.*

(*f*) *Section of Rubber Tubing*, to hold the Gooch crucible on the filter tube.

(*g*) *Drying Oven.*

(*h*) *Bunsen Burner.*

(*i*) *Suction Pump.*

(*j*) *Analytical Balance.*

(*k*) *Desiccator.*

Preparation of Sample

3. The sample shall be representative, and if it contains more than 2 per cent of water it shall be dehydrated by distillation in a copper still in accordance with the Standard Method of Test for Water in Creosote (A.S.T.M. Designation: D 370) of the American Society for Testing Materials,[3] and the water-free distillate returned to the residue. If the material is hard and brittle, it may be ground, and dried at a temperature below the temperature of volatilization of the material.

Preparation of Gooch Crucible

4. Insert the filter tube in the stopper of the filtering flask, set the Gooch crucible in the filter tube, and connect the flask to the suction pump. Fill the crucible with some of the suspension of asbestos in water, allow it partly to settle in the crucible, and apply a light

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] Prior to adoption as standard, this method was published as tentative from 1923 to 1927, being revised in 1926 and 1927.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

suction to draw off the water, leaving a firm mat of asbestos in the crucible. Add more suspended asbestos and repeat the process until a mat is built up that weighs 0.5 ± 0.1 g. after ignition (Note). Wash the asbestos mat thoroughly with water, dry in the oven, and ignite over a bunsen burner. Cool the crucible in the desiccator, weigh, and replace it in the dry filter tube supported in the clean, dry filtering flask.

NOTE.—In the determination, the asbestos apparently adsorbs irreversibly a small amount of soluble bitumen (usually 1 to 5 mg. per gram of asbestos) which is not removed by subsequent washing with solvent. The weight of asbestos used, therefore, should be kept within the specified limits to ensure reproducible results.

Procedure

5. (*a*) Weigh approximately 2 g. of the sample into a tared conical flask, and add in small portions 100 ml. of c.p. carbon tetrachloride with continued agitation until all lumps disappear and nothing adheres to the bottom of the flask. Then stopper the flask and set aside in subdued light for at least 12 hr.

(*b*) Decant the carbon tetrachloride solution carefully through the asbestos mat in the prepared Gooch crucible, with or without light suction as may be found necessary, retaining as much of the sediment as possible in the conical flask until the solution has drained through the mat. With a small amount of carbon tetrachloride wash down the sides of the flask and transfer the sediment and precipitate from the flask to the asbestos mat. The flask may be scrubbed with a feather if necessary in order to remove all of the precipitate. Wash the contents of the crucible with carbon tetrachloride until the washings are colorless, then apply suction to remove the carbon tetrachloride. Dry the crucible in the oven at 100 to 125 C. for 20 min., cool in the desiccator, and weigh. The increase in weight over the original weight is the weight of matter insoluble in carbon tetrachloride.

(*c*) If insoluble matter adheres to the flask, dry the flask, and weigh. Add the increase in weight over the original weight to the weight of insoluble matter in the Gooch crucible.

(*d*) In case there is any question involving the amount of mineral matter that may have passed through the filter, evaporate the filtrate, and burn the bituminous residue. If a carbonate mineral is present in the filtrate ash, add to the ash a few drops of ammonium carbonate solution, and dry at 100 C., then heat for a few minutes to a dull red heat, and cool in the desiccator. Weigh, and add the weight of ash obtained to the weight of matter insoluble in carbon tetrachloride.

Calculation and Report

6. (*a*) *Calculation.*—The weight of sample taken calculated to a water-free basis minus the total weight of matter insoluble in carbon tetrachloride is the weight of bitumen soluble in carbon tetrachloride. The percentage shall be calculated on the basis of water-free material.

(*b*) *Report.*—The proportion of bitumen soluble in carbon tetrachloride shall be reported on the basis of total bitumen taken as 100, as follows:

$$P = \frac{\text{bitumen soluble in carbon tetrachloride}}{\text{total bitumen}}$$

where:

P = proportion of bitumen soluble in carbon tetrachloride.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: A37.9-1943
AMERICAN ASSOCIATION STATE
HIGHWAY OFFICIALS STANDARD
A.A.S.H.O. NO.: T 52-42

Standard Method of Test for

DISTILLATION OF TAR PRODUCTS SUITABLE FOR ROAD TREATMENT[1]

A.S.T.M. Designation: D 20 – 30

ADOPTED, 1930.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 20; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

This method is presented without any definition of its scope and accuracy since it is believed that it will become applicable to a wider range of materials than indicated by the title. So far the committee has determined its accuracy on one class of materials and further work will be done.

Apparatus

1. The apparatus shall consist of the following:

(*a*) *Flask.*—A side-neck distillation flask, as shown in Fig. 1, having the following dimensions:

Diameter of bulb, outside	86 ± 1.5 mm.
Diameter of neck, inside	22 ± 1.0 mm.
Diameter of tubulature, inside	10 ± 0.5 mm.
Height of flask, outside	131 ± 1.5 mm.
Vertical distance bottom of bulb, outside, to horizontal tangent at tubulature inside	93 ± 1.5 mm.
Length of tubulature	220 ± 5.0 mm.
Angle of tubulature	73 ± 2 deg.
Thickness of tubulature wall	1.0 to 1.5 mm.

(*b*) *Condenser Tube.*—A suitable form of tapered glass condenser tube having the following dimensions:

Outside diameter of small end	12.5 ± 1.5 mm.
Outside diameter of large end	28.5 ± 3.0 mm.
Length	360.0 ± 4.0 mm.
Length of tapered part	100.0 ± 5.0 mm.

(*c*) *Shield.*—A galvanized iron shield, lined with $\frac{1}{8}$-in. asbestos, of the form and dimensions shown in Fig. 2, to protect the flask from air currents and to prevent radiation. The cover (top) may be of transite board made in two parts, or it may be of galvanized iron lined with $\frac{1}{8}$-in. asbestos.

NOTE.—The use of the shield with observation windows (see the Standard Method of Test for Distillation of Cut-Back Asphaltic Products (A.S.T.M. Designation: D 402) of the American Society for Testing Materials[3]) is permissible.

(*d*) *Receivers.*—Tared Erlenmeyer flasks having a capacity of 50 to 100 ml.

(*e*) *Thermometer.*—An A.S.T.M. High Distillation Thermometer, total immersion, graduated in either Centigrade or

[1] Under the standardization procedure of the Society this method is under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] Prior to its present adoption as standard, this method was published as tentative from 1911 to 1916, being revised in 1916. It was adopted in 1916, revised in 1918, but withdrawn and replaced by D 20 - 27 T which was published as tentative from 1926 to 1930, being revised in 1927, 1928, and 1930. Editorially revised in 1939 and 1942.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Fahrenheit degrees as specified, having a range of 0 to 400 C. or 30 to 760 F. and conforming to the requirements for this thermometer as prescribed in the Standard Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1) of the American Society for Testing Materials.[3]

NOTE.—Under certain test conditions the bulb of the thermometer may be 50 F. (28 C.) above the temperature indicated by the thermometer, and at an indicated temperature of 700 F. (371 C.) the temperature of the bulb is approaching a critical range in the glass. It is therefore not desirable to use this thermometer under such conditions at indicated temperatures above 700 F. (371 C.) without checking the ice point.

If water is present in excess of 2.0 per cent, the bituminous material shall be dehydrated before distillation in accordance with the Standard Method of Test for Water in Creosote (A.S.T.M. Designation: D 370) of the American Society for Testing Materials.[3]

Apparatus Assembly

4. (*a*) The flask shall be supported on a tripod or rings over two sheets of 20-mesh wire gauze, 150 mm. square, as shown in Fig. 3. It shall be connected to the condenser tube by a tight cork joint. The thermometer shall be inserted through a cork in the neck with the top of the bulb level with the lowest point of juncture of the tubulature and neck of the flask.

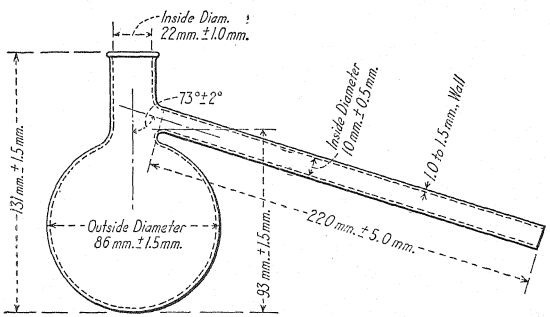


FIG. 1.—Distillation Flask.

Preparation of Sample

2. The sample, as received, shall be thoroughly stirred and agitated, warming, if necessary, to insure a complete mixture before the portion for analysis is removed.

Dehydration of Sample

3. The material may be tested for distillation without dehydration if water is present not to exceed 2.0 per cent.

(*b*) The axis of the bulb of the flask through the center of the neck shall be vertical and the thermometer shall be aligned on this axis.

(*c*) The distance from the neck of the flask to the outlet end of the condenser tube shall be not more than 600 nor less than 500 mm. The burner

should be protected from drafts by a suitable shield or chimney (see Fig. 3).

Procedure

5. (*a*) One hundred grams plus or minus 0.1 g. of the sample shall be weighed into the flask, the apparatus assembled, and heat applied so that the first drop comes over in from 5 to 15 min. The distillation shall be conducted at the rate of between 50 and 70 drops per min. and the distillate collected in weighed receivers. (For weighing the receivers and fractions, a balance accurate to at least 0.05 g. shall be used.) The condenser tube shall be warmed whenever necessary to prevent accumulation of solid distillates. The fraction shall be collected at the temperatures designated by the specifications. The receivers shall be changed when the thermometer indicates the maximum temperature for each fraction. When the maximum specified temperature of the test is indicated by the thermometer, the flame shall be removed and any oil which has condensed in the condenser tube shall be drained into the last fraction.

(*b*) The residue shall be retained in the flask with the cork and thermometer in position until no vapors are visible, and it shall then be weighed. If tests of the residue are required, the flask

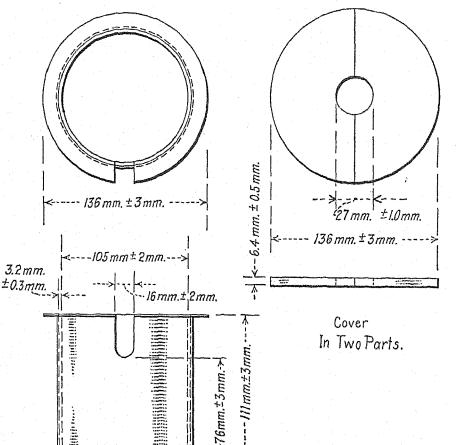


FIG. 2.—Shield.

shall then be inclined so that the residue will flow around the sides, thus collecting any condensed vapors that may be on the sides of the flask, after which the residue shall be poured into a suitable receptacle and covered. If the residue becomes so cool that it cannot be poured readily from the flask, it shall be reheated to a temperature not exceeding 125 C. by holding the bulb of the flask in a suitable bath and not by the application of flame.

(*c*) During the progress of the distillation the thermometer shall remain in its original position. No correction shall be made for the emergent stem of the thermometer.

Report

6. The results of the distillation test shall be reported in percentages by weight of water-free material. The following fractions are usually reported:

Up to 170 C.
170 to 235 C.
235 to 270 C.
270 to 300 C.
Residue.

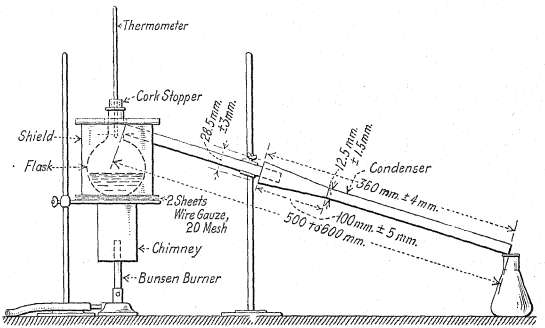


FIG. 3—Distillation Apparatus Assembly.

Standard Method of Test for

DISTILLATION OF CUT-BACK ASPHALTIC PRODUCTS[1]

A.S.T.M. Designation: D 402 - 36

ADOPTED, 1936.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 402; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test is intended for the separation of the volatile and nonvolatile portions of cut-back asphaltic products.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Flask.*—A side neck distillation flask, as shown in Fig. 1, having the following dimensions:

Diameter of bulb, outside.....	86 ± 1.5 mm.
Diameter of neck, inside.....	22 ± 1.0 mm.
Diameter of tubulature, inside.	10 ± 0.5 mm.
Height of flask, outside.....	131 ± 1.5 mm.
Vertical distance bottom of bulb, outside, to horizontal tangent at tubulature, inside.	93 ± 1.5 mm.
Length of tubulature.....	220 ± 5.0 mm.
Angle of tubulature.....	73 ± 2 deg.
Thickness of tubulature wall..	1.0 to 1.5 mm.

(*b*) *Condenser.*—A 250-mm. standard glass-jacketed condenser. The following dimensions are recommended (see Fig. 3):

Length of jacket excluding the necks.....	250 ± 5 mm.
Outside diameter of adapter of condenser tube.....	23 ± 1 mm.
Length of adapter.....	75 ± 5 mm.
Outside diameter of condenser tube proper.....	12.5 ± 0.5 mm.
Over-all length of condenser tube including adapter....	475 ± 25 mm.

(*c*) *Adapter.*—An adapter of the curved design having a heavy wall (1 mm.) and reinforced top glass, with an angle of approximately 105 deg., and with a diameter at the large end of approximately 18 mm. The outlet end shall be ground to an angle of 45 ± 5 deg. with the inside vertical.

(*d*) *Shield.*—A galvanized iron shield, lined with $\frac{1}{8}$-in. asbestos, fitted with transparent covered windows, of the form and dimensions shown in Fig. 2, used to protect the flask from air currents and to prevent radiation. The cover (top) may be of transite board made in two parts, or it may be of galvanized iron lined with $\frac{1}{8}$-in. asbestos.

(*e*) *Receivers.*—Graduated cylinders, of uniform diameter, with a pressed or molded base and a lipped top. The over-all height shall be not less than

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] Prior to adoption as standard, this method was published as tentative from 1934 to 1936, being revised in 1936. Editorially revised and rearranged in 1939.

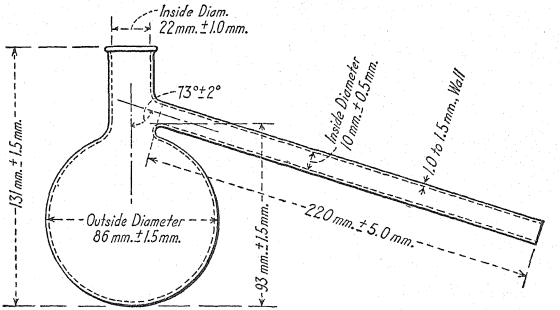


FIG. 1.—Distillation Flask.

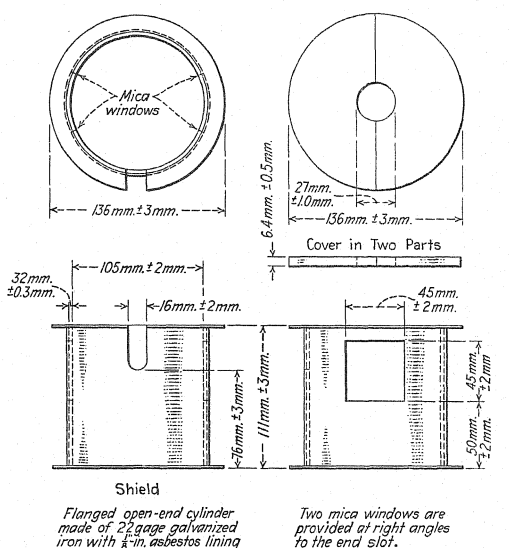


FIG. 2.—Shield.

24.8 cm. (9¾ in.) nor more than 26.0 cm. (10¼ in.). The cylinder shall be graduated in single milliliters to contain 100 ml., and the graduated portion shall be not less than 17.78 cm. (7 in.) nor more than 20.32 cm. (8 in.) in length. Each fifth graduation shall be distinguished by a longer line, and the graduations shall be numbered from the bottom up at intervals of 10 ml. The graduations shall not be in error by more than 1 ml. at any point on the scale.

(*f*) *Thermometer.*—An A.S.T.M. High Distillation Thermometer, total immersion, graduated in either Centigrade or Fahrenheit degrees as specified, having a range of 0 to 400 C. or 30 to 760 F. and conforming to the requirements for this thermometer as prescribed in the Standard Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1) of the American Society for Testing Materials.[3]

Preparation of Sample

3. The sample, as received, shall be thoroughly stirred and agitated, warming, if necessary, to insure a complete mixture before the portion for analysis is removed.

Dehydration of Sample

4. The material may be tested for distillation without dehydration if water is present not to exceed 2.0 per cent. If water is present in excess of 2.0 per cent, the bituminous material shall be dehydrated before distillation in accordance with the Standard Method of Test for Water in Creosote (A.S.T.M. Designation: D 370) of the American Society for Testing Materials.[3]

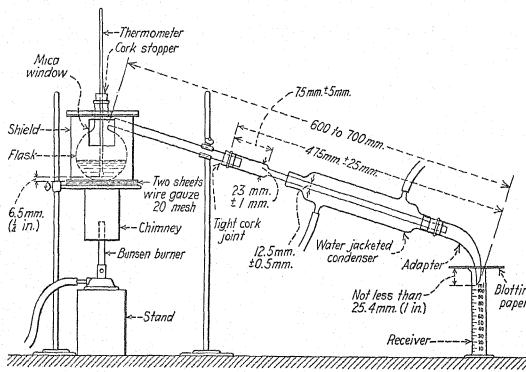


FIG. 3.—Distillation Apparatus Assembly.

Apparatus Assembly

5. (*a*) The flask shall be supported on a tripod or ring over two sheets of 20-mesh wire gauze, 150 mm. square as shown in Fig. 3. It shall be connected to the condenser tube by a tight cork joint. The thermometer shall be inserted through a cork in the neck with the bottom of the bulb 6.5 mm. (¼ in.) from the bottom of the flask.

(*b*) The axis of the bulb of the flask through the center of the neck shall be vertical and the thermometer shall be aligned on this axis.

(*c*) The distance from the neck of the

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

flask to the outlet end of the adapter shall be not more than 700 nor less than 600 mm. The burner should be protected from drafts by a suitable shield or chimney (see Fig. 3).

(*d*) The adapter shall be adjusted over the end of the condenser tube so as to conduct the distillate into the receiver, and the top of the receiver shall be covered closely during the distillation with a piece of blotting paper or its equivalent, which shall be cut so as to fit the adapter tightly. The adapter shall extend into the receiver at least 2.54 cm. (1 in.) but not below the 100-ml. mark. Unless the laboratory air temperature is between 12.8 and 18.3 C. (55 and 65 F.) the receiver shall be immersed up to the 100-ml. mark in a transparent bath maintained between these temperatures.

(*e*) The condenser tube shall be clean and dry.

Procedure

6. (*a*) Two hundred milliliters[4] (calculated from the specific gravity of the material at 15.5 C. (60 F.)) of the sample shall be weighed into the flask, the apparatus assembled and heat applied so that the first drop comes over in from 5 to 15 min. The distillation shall be conducted at the rate of between 50 and 70 drops per min., except that near the end of the distillation the heat input shall not be so rapid as to result in a temperature in excess of 360 C. (680 F.) after the flame has been removed. Should the sample foam, the distillation rate will have to be reduced, but the normal rate shall be resumed as soon as possible. If excess foaming persists, the distillation may be more easily controlled by applying the flame near the edge of the bulb instead of at the center of same. The distillate shall be collected in the specified receivers, and the volume of distillate at all specified temperatures recorded. The volume of any separated water shall also be recorded. When the maximum specified temperature of the test is indicated by the thermometer, the flame shall be removed and the residue poured *immediately* into a 6-oz. tin box, 70 mm. ($2\frac{3}{4}$ in.) in diameter and 45 mm. ($1\frac{3}{4}$ in.) in depth (Note), placed on its cover to prevent too rapid cooling at the bottom. Any oil which may remain in the condenser tube shall be drained into the last receiver.

NOTE.—Containers known in the drug trade as seamless "ointment boxes" may be obtained in dimensions conforming to the above requirements.

(*b*) As soon as no further vaporization is apparent, the residue shall be stirred to insure uniformity, and then poured into the necessary apparatus for the required tests.

(*c*) During the progress of the distillation the thermometer shall remain in its original position. No correction shall be made for the emergent stem of the thermometer. Temperatures to be observed in the distillation test shall be corrected for the effect of the altitude of the laboratory in which the test is made in accordance with the Standard Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (A.S.T.M. Designation: D 86) of the American Society for Testing Materials.[3]

Report

7. The results of the distillation test shall be reported in percentage by volume of water-free material. The following fractions are usually reported:

Up to 225 C. (437 F.),
Up to 316 C. (600 F.), and
Up to 360 C. (680 F.).

Additional fractions may be specified, such as:

Up to 160 C. (320 F.),
Up to 175 C. (347 F.), and
Up to 190 C. (374 F.).

[4] In the case of persistently foaming material, one half of this amount may be employed.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: A37.11-1944
AMERICAN ASSOCIATION STATE
HIGHWAY OFFICIALS STANDARD
A.A.S.H.O. NO.: T 51-44

Standard Method of Test for
DUCTILITY OF BITUMINOUS MATERIALS[1]

A.S.T.M. Designation: D 113 - 44

ADOPTED, 1935; REVISED, 1939, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 113; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. The ductility of a bituminous material is measured by the distance to which it will elongate before breaking when two ends of a briquet specimen of the material, of the form described in Section 2, are pulled apart at a specified speed and at a specified temperature. Unless otherwise specified, the test shall be made at a temperature of 25 ± 0.5 C. (77 ± 0.9 F.) and with a speed of 5 cm. per min. plus or minus 5.0 per cent. When a low-temperature ductility test is desired the test shall be made at a temperature of 4 C. (39.2 F.) and at a rate of pull of 1 cm. per min.

Apparatus

2. (*a*) *Mold.*—The mold shall be similar in design to that shown in Fig. 1. Dimensions shown in Fig. 1 shall be as given with the permissible variations indicated. The mold shall be made of brass, the ends *b* and *b'* being known as clips, and the parts *a* and *a'* as sides of the mold. The dimensions of the mold shall be such that, when properly assembled, it will form a briquet specimen having the following dimensions:

Total length	7.45 to 7.55 cm.
Distance between clips	2.97 to 3.03 cm.
Width at mouth of clip	1.98 to 2.02 cm.
Width at minimum cross-section (halfway between clips)	0.99 to 1.01 cm.
Thickness throughout	0.99 to 1.01 cm.

(*b*) *Water Bath.*—The water bath shall be maintained at the specified test temperature, varying not more than 0.1 C. (0.18 F.) from this temperature. The volume of water shall be not less than 10 liters, and the specimen shall be immersed to a depth of not less than 10 cm. and shall be supported on a perforated shelf not less than 5 cm. from the bottom of the bath.

(*c*) *Testing Machine.*—For pulling the briquet of bituminous material apart, any apparatus may be used which is so constructed that the specimen will be continuously immersed in water as specified in Section 3(*c*), while the two clips are pulled apart at a uniform speed, as specified, without undue vibration.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] Prior to adoption as standard, this method was published as tentative from 1921 to 1935, being revised in 1922, 1926, 1932, and 1935.

Procedure

3. (*a*) *Molding Test Specimen.*—The bituminous material to be tested shall be completely melted until thoroughly fluid by heating it in an oil bath maintained at the minimum temperature needed to properly liquefy the sample (Note). It shall then be strained through a No. 50 (297-micron) sieve conforming to the requirements prescribed in the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials[3] and, after a thorough stirring, poured into the mold. The mold shall be assembled on a brass plate and, so as to prevent the material under test from sticking, the surface of the plate and interior surfaces of the sides *a* and *a'*, Fig. 1, of the mold shall be

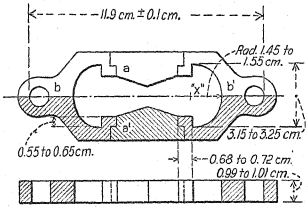


FIG. 1.—Mold for Ductility Test Specimens.

NOTE.—The opening in the end of each clip, as indicated by "x," shall be half an ellipse having a transverse axis of 3.2 ±0.05 cm. and half of the longitudinal axis shall be 1.45 to 1.55 cm.

thoroughly amalgamated. The plate upon which the mold is placed shall be perfectly flat and level so that the bottom surface of the mold will touch it throughout. In filling the mold, care shall be taken not to disarrange the parts and thus distort the briquet. In filling, the material shall be poured in a thin stream back and forth from end to end of the mold until it is more than level full. It shall be left to cool to room temperature for a period of from 30 to 40 min. and then placed in the water bath maintained at the specified temperature of test for 30 min., after which the excess bitumen shall be cut off by means of a hot straight-edged putty knife or spatula so that the mold shall be just level full.

NOTE.—When paving asphalt cements are being tested, the oil bath shall be maintained at a temperature of from 150 to 160 C. (302 to 320 F.).

(*b*) *Keeping Specimen at Standard Temperature.*—The brass plate and mold, with briquet specimen, shall then be placed in the water bath and kept at the specified temperature for a period of from 85 to 95 min., when the briquet shall be removed from the plate, the side pieces detached, and the briquet immediately tested.

(*c*) *Testing.*—The rings at each end of the clips shall be attached to the pins or hooks in the testing machine and the two clips pulled apart at a uniform speed as specified until the briquet ruptures. A variation of plus or minus 5 per cent from the speed specified will be permissible. The distance through which the clips have been pulled to produce rupture shall then be measured in centimeters. While the test is being made, the water in the tank of the testing machine shall cover the specimen both above and below it by at least 2.5 cm. and shall be kept continuously at the temperature specified within plus or minus 0.5 C. (0.9 F.).

Report

4. (*a*) A normal test is one in which the material between the two clips pulls out to a point or thread until rupture occurs at the point where the thread has practically no cross-sectional area. The average of three normal tests shall be taken and reported as the ductility of the sample.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(*b*) If the bituminous material comes in contact with the surface of the water or the bottom of the bath, the test shall not be considered normal. The specific gravity of the bath shall be adjusted by the addition of either methyl alcohol or sodium chloride so that the bituminous material does not either come to the surface of the water, or touch the bottom of the bath at any time during the test.

(*c*) If a normal test is not obtainable on three successive tests, the ductility shall be reported as being unobtainable under the conditions of the test.

Standard Methods of

TESTING EMULSIFIED ASPHALTS[1]

A.S.T.M. Designation: D 244 - 42

ADOPTED, 1939; REVISED, 1940, 1942.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 244; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods are intended for the examination of asphalt emulsions composed, principally, of a semisolid or liquid asphaltic base, water, and an emulsifying agent.

VISCOSITY

2. The viscosity of emulsified asphalt shall be determined by means of the Saybolt Furol viscosimeter.

Apparatus

3. The apparatus shall consist of the following:

(*a*) *Viscosimeter.*—A Saybolt Furol viscosimeter conforming to the requirements specified in the Standard Method of Test for Viscosity by Means of the Saybolt Viscosimeter (A.S.T.M. Designation: D 88) of the American Society for Testing Materials.[3]

(*b*) *Strainer.*—A 20-mesh strainer of iron wire cloth, framed or unframed.

Procedure

4. (*a*) The viscosity determination shall be made at 77 F. (25 C.) and shall be expressed in seconds, Saybolt Furol, being the time in seconds for the delivery of 60 ml. of emulsion.

(*b*) While the Saybolt Furol viscosimeter is not used for petroleum products and lubricants when the time of flow is less than 25 sec., this instrument is satisfactory for testing emulsified asphalt when the time of flow is not less than 20 sec.

(*c*) *Preparation of Sample.*—The sample shall be stirred thoroughly without incorporating bubbles in it and then poured into a 4-oz. bottle. The bottle shall then be placed in the water bath at 77 F. (25 C.) for 30 min. and the sample then mixed in the bottle by inverting several times, slowly enough to prevent bubble formation. The sample shall then be poured into the vis-

[1] Under the standardization procedure of the Society, these methods are under the joint jurisdiction of A.S.T.M. Committee D-4 on Road and Paving Materials and Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] Prior to adoption as standard, these methods were published as tentative from 1926 to 1939, being revised in 1928, 1933, 1934, 1935, 1936, and 1939.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

cosity tube through a 20-mesh strainer, allowing a small portion to flow through the outlet tube to waste. The cork shall then be placed in position, the tube filled and without again stirring the sample the viscosity shall be determined as described in the Standard Method of Test for Viscosity by Means of the Saybolt Viscosimeter (A.S.T.M. Designation: D 88) of the American Society for Testing Materials.[3]

ring burners are required, one approximately 6 in. in inside diameter with the holes bored on the inside periphery, and one approximately 2 in. in diameter with the holes bored on top.

(*b*) *Connecting Apparatus.*—A connecting tube, tin shield, condenser trough, condenser tube and graduated cylinder, as shown in Fig. 1.

(*c*) *Thermometer.*—An A.S.T.M. Low-Distillation Thermometer, graduated in either Fahrenheit or Centigrade degrees

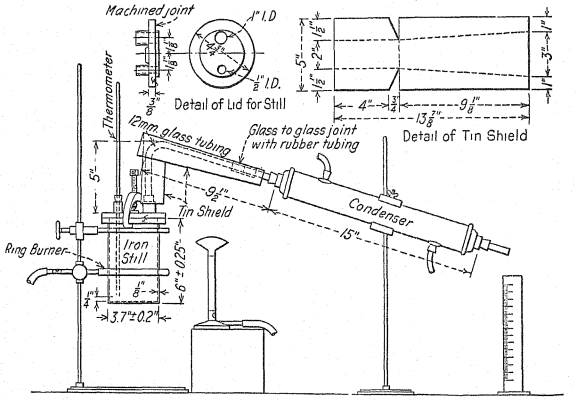


FIG. 1.—Apparatus Assembly for Distillation Test of Emulsified Asphalts.

DISTILLATION

Apparatus

5. The apparatus shall consist of the following:

(*a*) *Iron Still.*—The iron still shall be approximately 6 by 3½ in. in inside diameter with adjustable ring burner having holes on the inner periphery to fit around the outside of the still.

NOTE.—A modification of this still is shown in Fig. 2; either the still shown in Fig. 1 or that shown in Fig. 2 may be used interchangeably. It consists of the regular still with an expansion chamber superimposed thereon. Two additional

as specified, having a range of 30 to 580 F. or 0 to 300 C. and conforming to the requirements for this thermometer as prescribed in the Standard Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1) of the American Society for Testing Materials.[3]

NOTE.—The details of the assembly of apparatus for the distillation test are illustrated in Fig. 1.

Procedure

6. (*a*) Exactly 200 g. of a well-mixed and representative sample of the emul-

sion shall be placed in the previously weighed iron still (including lid, clamp, thermometer, and gasket, if gasket is used).

(*b*) A gasket of oiled paper may be used between the still and its cover or the joint ground to a tight fit. The cover shall be clamped securely on the still.

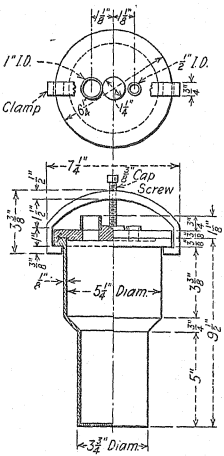


FIG. 2.—Iron Still for Use with Badly Foaming Emulsions.

(*c*) The thermometer shall be inserted through the small hole in the cover, using a cork stopper, so that the end of the bulb is ½ in. from the bottom of the still.

(*d*) The ring burner shall be placed around the still and the heat applied by this means to the top of the still. Just enough heat from a bunsen burner shall also be applied to the connecting tube to prevent condensation of water in this tube.

(*e*) After practically all the condensate has been removed from the still and the temperature of the residue has reached 250 F. (121 C.), the position of the heat from the ring burner shall be lowered to midway of the still and held there until the thermometer reaches 349 F. (176 C.). The burner shall then be rapidly lowered to within ¼ in. of the bottom of the still and the temperature increased to and maintained at 500 F. (260 C.) for 15 min. This latter period of heating is necessary to insure a smooth homogeneous residue in the still.

(*f*) At the expiration of the heating period at the maximum temperature, the still and accessories shall again be weighed as described in Paragraph (*a*) and the percentage residue calculated and reported. The cover shall then be removed from the still and suitable portions of the residue shall be poured immediately through a 297-micron (No. 50) sieve into suitable molds and containers for making the required tests. The residue in the molds and containers shall be permitted to cool, uncovered, to laboratory room temperature and thereafter tested as described in Sections 36 to 40, inclusive.

NOTE.—When it appears impossible to distill an emulsified asphalt in the still described in Section 5 (*a*) due to excessive foaming of the emulsion, then the modified still shown in Fig. 2 should be substituted for the still shown in Fig. 1 and the following procedure followed: Place the 6-in. burner around the larger diameter of the still near its top. This serves as a support. Place the 4-in. burner immediately beneath the flare and the 2-in. burner not less than 2 in. below the bottom. Distillation is started with only the 2-in. burner lighted. Practically all of the distillate should be over in about 45 min. When the distillation apparently stops, light the two larger ring burners and adjust to a low flame. Distillation resumes and when it stops again increase the heat by adjusting the flame of the 2-in. burner. When the temperature can be read upon the thermometer, increase the rate of heating by raising the flame on both the 2 and 4-in. burners and bring the temperature to 500 F. (260 C.).

If any evidence is noted of the emulsion beginning to foam over in the delivery tube, re-

move the 2-in. burner quickly and raise a pan of water so as to immerse the still bottom to a depth of about 2 in. for a moment which will check the foaming. Upon resumption of heating, watch delivery tube carefully and repeat treatment if necessary.

When the residue has reached and remained at 500 F. (260 C.) for 15 min., proceed as described in Section 6 for the regular still. While the distillation should be completed in not less than 1 hr. nor more than 1½ hr. from the first application of heat to the still, the maximum stated is not mandatory, as a longer time may be required in some cases to avoid foaming of the emulsion into the condenser.

If the residue in the still prior to pouring the ductility and penetration specimens appears granular or heterogeneous in any way, stir with a spatula until the material runs from the spatula in strings instead of drops, and then pour.

DETERMINATION OF WATER

Scope

7. This method of test determines water existing in a sample of bituminous emulsion by distilling the sample with a volatile solvent.

Apparatus

8. (a) The apparatus shall consist of a metal still or glass flask, heated by suitable means and provided with a reflux condenser discharging into a trap connected to the still or flask. The trap serves to collect and measure the condensed water and to return the solvent to the still. The type of distilling apparatus used is not an essential feature of this method.

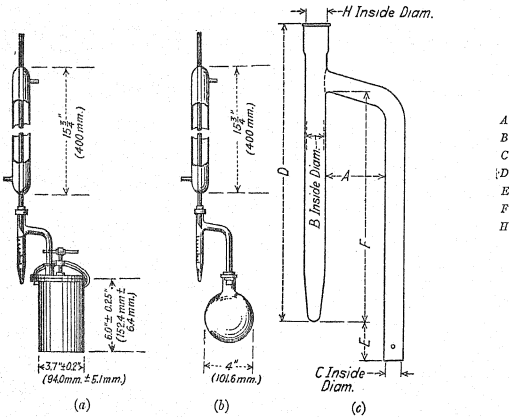


FIG. 3.—Apparatus for Determining Water.

(b) *Metal Still.*—The metal still, Fig. 3 (*a*), shall be a vertical cylindrical vessel, preferably of copper, having a faced flange at the top to which the head is tightly attached by means of a clamp. The head shall be of metal, preferably of brass or copper, and be provided with a tubulation 1 in. in inside diameter.

(c) *Glass Still.*—The glass flask,

Fig. 3 (*b*), shall be of the short neck, round-bottom type, made of well-annealed glass, having an approximate capacity of 500 ml.

(*d*) *Heat Source.*—The burner used with the metal still shall be a ring gas burner 100 mm. (4 in.) in inside diameter. With the glass flask, an ordinary gas burner or electric heater may be used as the source of heat.

(*e*) *Condenser.*—The condenser shall be of the water-cooled, reflux, glass-tube type, having a condenser jacket not less than 400 mm. (15¾ in.) in length with an inner tube 9.5 to 12.7 mm. (⅜ to ½ in.) in outside diameter. The end of the condenser to be inserted in the trap shall be ground off at an angle of 30 ± 5 deg. from the vertical axis of the condenser.

(*f*) *Trap.*—The trap shall be made of well-annealed glass, constructed in accordance with Fig. 3 (*c*), and shall be graduated from 0 to 2 ml. in 0.1-ml. divisions, and from 2 to 25 ml. in 0.2-ml. divisions. The tolerance of the graduations between 0 and 2 ml. shall be plus or minus 0.05 ml. and between 2 and 25 ml. shall be plus or minus 0.1 ml.

Solvent

9. The solvent used when testing bituminous emulsions shall be a coal-tar naphtha or a light oil and shall conform to the following distillation requirements, determined in accordance with the Standard Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (A.S.T.M. Designation: D 86) of the American Society for Testing Materials:[3]

> 98 per cent shall distill between 248 F. (120 C.) and 482 F. (250 C.).

Sample

10. The sample shall be thoroughly representative of the material to be tested and the portion of the sample used for the test shall be thoroughly representative of the sample itself. Deviation from this requirement shall not be permitted.

NOTE.—The difficulties in obtaining proper representative samples for this determination are unusually great so that the importance of sampling cannot be too strongly emphasized.

Procedure

11. (*a*) When the material to be tested contains less than 25 per cent of water, exactly a 100-g. sample shall be placed in the still or flask. When the material contains more than 25 per cent of water, the sample shall be 50 g. The sample to be tested shall be thoroughly mixed with an equal volume of solvent by swirling, proper care being taken to avoid any loss of material.

(*b*) The connections between the still or flask, trap and condenser shall be made by means of tight-fitting corks as shown in Fig. 3 (*a*) and (*b*). The end of the condenser inserted in the trap shall be adjusted to that position which will allow the end to be submerged to a depth of not more than 1 mm. (0.04 in.) below the surface of the liquid in the trap after distillation conditions have been established. When the metal still is used, a heavy paper gasket moistened with the solvent shall be inserted between the lid and flange before attaching the clamp. A loose cotton plug shall be inserted in the top of the condenser tube to prevent condensation of atmospheric moisture in the condenser tube.

(*c*) Heat shall then be applied and so regulated that the condensed distillate falls from the end of the condenser at the rate of from 2 to 5 drops per sec. The ring burner used with the metal still shall be placed about 3 in. above the bottom of the still at the beginning of the distillation and gradually lowered as the distillation proceeds.

(*d*) The distillation shall be continued at the specified rate until no water is visible on any part of the apparatus except at the bottom of the trap. This operation usually requires less than 1 hr. A persistent ring of condensed water in the condenser tube shall be removed by increasing the rate of distillation for a few minutes.

Calculation and Reporting of Results

12. The volume of condensed water measured in the trap at room temperature multiplied by 100 and divided by the weight of sample used, shall be the percentage of water and shall be reported as "....per cent water by weight, A.S.T.M. Method D 244."

Reproducibility of Results

13. The accuracy to be expected with this method is that duplicate determinations of water should not differ from each other by more than one division on the trap.

SETTLEMENT

Apparatus

14. The apparatus shall consist of the following:

(*a*) *Cylinders.*—Two glass cylinders of 500-ml. capacity with pressed or molded glass base and cork or glass stoppers. The outside diameter shall be 5.0 ± 0.5 cm. and the cylinders shall be graduated at each 5-ml. interval to the 500-ml. mark.

(*b*) *Glass Pipette.*—A siphon, glass tube pipette, 60-ml. capacity, form optional.

Procedure

15. (*a*) A 500-ml. sample, representative of the emulsion, shall be placed in each of two glass cylinders. The cylinder shall be stoppered airtight and stood aside unmolested, at laboratory air temperature for 5 days. After standing for this 5-day period, approximately the first 55 ml. of emulsion shall be removed by means of the pipette or siphon from the top of each cylinder without disturbing the balance of their contents. Exactly 50 g. of each of the two samples, after each has been thoroughly mixed separately, shall be weighed into separate 600-ml. low-form glass beakers and the asphaltic residue determined by evaporation at 325 F. (163 C.) for 3 hr. in the apparatus described in the Tentative Method of Test for Loss on Heating of Oil and Asphaltic Compounds (A.S.T.M. Designation: D 6) of the American Society for Testing Materials.[3]

(*b*) After removal of the first sample, approximately the next 390 ml. shall be siphoned off from each of the cylinders. The residue remaining in the cylinders shall be mixed thoroughly and exactly 50 g. shall be weighed out from each of them and the amount of asphaltic residue (all sediment, if any, included) shall be determined by evaporation as described in Paragraph (*a*) for the two top samples.

(*c*) The numerical difference between the average percentage of asphaltic residue from the two top samples and the average percentage found in the two bottom samples shall be recorded.

DEMULSIBILITY

Apparatus and Reagents

16. The apparatus and reagents shall consist of the following:

(*a*) *Wire Cloth.*—Three pieces of 1410-micron (No. 14) iron wire cloth approximately 5 in. square, unframed, having wire diameters and openings which conform to the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials.[3]

(*b*) *Beakers.*—Three metal beakers of 600-ml. capacity each.

(*c*) *Rods.*—Three metal rods, rounded ends, approximately $\frac{5}{16}$ in. in diameter.

(*d*) *Burette.*—A 50-ml. glass burette graduated in 0.1 ml.

(*e*) *Calcium Chloride Solution (0.02 N).*

(*f*) *Calcium Chloride Solution (0.10 N).*

Procedure

17. (*a*) The percentage of residue shall be determined by distillation as described in Section 6.

(*b*) The weight of each assembly of beaker, rod, and wire cloth shall be recorded.

(*c*) Exactly 100 g. of the emulsified asphalt shall be weighed into each of three 600-ml. tared beakers. Over a period of approximately 2 min., 35 ml. of 0.02 *N* $CaCl_2$ solution (if quick-setting emulsion is being tested) or 50 ml. of 0.10 *N* $CaCl_2$ solution (if mixing type emulsion is being tested) shall be added to each beaker from a burette. While adding the solution of $CaCl_2$, the contents of the beaker shall be stirred continuously and vigorously, kneading lumps against the sides of the beaker to insure thorough mixing of the reagent with the emulsion. This operation shall be performed after bringing the weighed sample of emulsion and the reagent to the standard temperature of 77 ± 1.0 F. (25 ± 0.5 C.).

(*d*) One of the wire cloths shall be fitted over a beaker or other suitable vessel and the mixture of emulsion and reagent poured through the wire cloth. The beaker, containing the sample and metal rod, shall be rinsed with distilled water. All lumps shall be kneaded and broken up and the washing of the beaker, rod, and wire cloth shall be continued until there is no longer any appreciable color imparted to the wash water. After washing as directed, the beaker, rod, and wire cloth used in each individual test shall be placed in a drying oven and dried at 325 F. (163 C.) to constant weight.

Calculation

18. The total weight thus obtained less the total tare weight of the beaker, rod and wire cloth shall be the weight of the residue by the demulsibility test. The ratio of the average weight in grams from three tests of each individual sample of emulsified asphalt, *A*, to the weight in grams of residue per 100 g. of emulsion, *B*, obtained in the test for residue by distillation described in Section 6 multiplied by 100, shall be recorded as the percentage of demulsibility of the sample tested:

$$\text{Percentage demulsibility} = \frac{A}{B} \times 100$$

SIEVE TEST

Apparatus and Reagents

19. The apparatus and reagents shall consist of the following:

(*a*) *Sieve.*—A sieve having a 3-in. frame conforming to Section 4 (*a*) and (*b*) of the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials,[3] and having 840-micron (No. 20) wire sieve cloth conforming to Section 3 of these specifications.

(*b*) *Pan.*—A tin box cover or shallow metal pan of appropriate size to fit over the bottom of the standard sieve.

(*c*) *Sodium Oleate Solution (2 per cent).*—Prepare a 2 per cent solution of pure sodium oleate in distilled water.

Procedure

20. The weight of the sieve and pan shall be recorded and the wire cloth of the No. 20 sieve shall then be wet with the sodium oleate solution (2 per cent). Exactly 1000 g. of the emulsified asphalt

shall then be weighed and poured through the wire sieve, the container and the residue on the sieve being washed thoroughly with the sodium oleate solution until the washings run clear. The pan shall then be placed under the sieve and heated for 2 hr. in a drying oven whose interior temperature is 220 F. (105 C.), then cooled in a desiccator, and weighed.

Calculation

21. The total weight of the sieve, pan, and residue in grams less the combined tare weight of the sieve and pan, is the weight of the residue by the sieve test. The percentage of residue retained on the sieve shall be calculated from this weight.

MISCIBILITY WITH WATER

NOTE.—This test is not applicable to the so-called quick-setting type of emulsions.

Procedure

22. To about 50 ml. of the emulsion shall be gradually added about 150 ml. of distilled water, stirring the mixture while adding the water. The temperature is not important but should be between 21 and 25 C. (70 and 77 F.). The mixture shall be allowed to stand for 2 hr. and then examined for any appreciable coagulation of the asphalt content of the emulsion.

MODIFIED MISCIBILITY WITH WATER

Scope

23. This method of test covers the procedure for determining the miscibility with water of medium-setting and slow-setting types of asphalt emulsions. This test is not applicable to the quick-setting type of asphalt emulsions.

Apparatus

24. The apparatus shall consist of the following:

(*a*) *Graduate.*—A 50-ml. graduated cylinder.

(*b*) *Beaker.*—A 400-ml. Griffin low-form glass beaker.

(*c*) *Glass Tubes.*—Three glass tubes, 7 mm. in outside diameter, 5 mm. in inside diameter, and 15 cm. in length, fitted with suitably bored No. 8 corks, adjusted as described in Section 25.

(*d*) *Supporting Strip.*—A strip of metal or wood, approximately 15 cm. in length, 2.5 cm. in width, and 0.5 cm. in thickness, with a hole 10 mm. in diameter in the center.

(*e*) *Crucibles.*—Three 15 or 25-ml. porcelain crucibles, or three 30-ml. beakers of heat-resistant glass.[4]

(*f*) *Oven.*—A constant temperature oven as described in the Tentative Method of Test for Loss on Heating of Oil and Asphaltic Compounds (A.S.T.M. Designation: D 6) of the American Society for Testing Materials.[3]

(*g*) *Balance.*—An analytical balance accurate to 0.1 mg.

Assembly of Apparatus

25. Adjust the position of the corks on the glass tubes by measuring 200 ml. of distilled water at 20 to 25 C. into the 400-ml. beaker, placing the supporting strip across the top of the beaker, inserting a tube through the hole, and adjusting the position of the cork so that when the tube is supported by the cork resting on the supporting strip, the lower end of the tube is immersed in the water to a depth of 1 cm. below the surface. In the same manner, adjust the second and third tubes so that the depth of immersion is 2.5 and 4.6 cm., respectively.

[4] Pyrex glass is very satisfactory for this purpose.

NOTE.—Due to slight differences in height and diameter of 400-ml. beakers as obtained commercially, it may be necessary to readjust the tubes when used in different beakers. In any event, the third or bottom tube shall project into the emulsion so that the tip is within 1 to 1.5 mm. of the bottom of the beaker.

Procedure

26. (*a*) Measure 50 ml. of the emulsion at a temperature of 20 to 25 C. into the graduated cylinder and transfer to the 400-ml. beaker. Wash the graduate with three 50-ml. portions of distilled water at 20 to 25 C. and add the washings to the beaker, bringing the final volume to 200 ml. Stir the emulsion and water with a glass rod until uniformly mixed, cover the beaker with a watch glass, and allow the mixture to stand undisturbed for 2 hr.

(*b*) Weigh the three crucibles or 30-ml. beakers, and a watch glass for each, to the nearest 0.1 mg. After the diluted emulsion has stood for 2 hrs., remove the watch glass and place the supporting strip across the top of the 400-ml. beaker. Take a sample of approximately 1 g. from the top layer and transfer to one of the crucibles or beakers, using the first or 1-cm. depth tube as a pipette. Close the top of the tube with the finger, insert the tube to the proper depth, remove the finger while the emulsion rises in the tube, and then replace the finger on top of the tube so that when the tube is removed its contents of emulsion will be pipetted from the beaker. After removal, wipe off the adhering liquid on the outside of the tube with filter paper before transferring the sample to the crucible. In like manner, take samples from the middle and bottom of the diluted emulsion, using the second and third tubes, respectively. Weigh the crucibles with their accompanying samples of emulsion, and determine the weight of each of the three samples by difference. Cover the crucibles with watch glasses to retard evaporation.

(*c*) Remove the watch glasses from the crucibles and place the samples in the oven at 163 C. for 2 hr., then remove, cool, and weigh.

Calculations and Report

27. Calculate the percentage of asphalt residue in the top, middle, and bottom levels. Report the maximum numerical difference in percentage of asphalt content between any two of the three levels.

CEMENT MIXING

Scope

28. This method of test is intended for determining the amount of coagulation when portland cement is mixed with the slow-setting type of asphalt emulsions for fine aggregate mixes. This test is not applicable when 2 per cent or more coagulation is anticipated.

Apparatus

29. The apparatus shall consist of the following:

(*a*) *Sieves.*—A 177-micron (No. 80) sieve and a 1410-micron (No. 14) sieve, made of iron wire cloth having wire diameters and openings conforming to the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials.[3]

(*b*) *Dish.*—A round-bottom iron dish or a kitchen saucepan of approximately 500-ml. capacity.

(*c*) *Stirring Rod.*—A steel rod with rounded ends, approximately $\frac{1}{2}$ in. in diameter.

(*d*) *Graduate.*—A 100-ml. graduated cylinder.

Cement

30. The high-early-strength portland cement used in the test shall conform to

the requirements for type III of the Standard Specifications for Portland Cement (A.S.T.M. Designation: C 150) of the American Society for Testing Materials,[2] and shall have a minimum specific surface area of 1900 sq. cm. per gram.

Procedure

31. (*a*) Dilute the emulsion to be tested with distilled water to a residue of 55 per cent as determined by either distillation, or evaporation for 3 hr. at 163 C.

(*b*) Sieve a portion of the cement through the 177-micron (No. 80) sieve. Weigh 50 g. of the cement passing the 177-micron sieve into the iron dish.

(*c*) Add 100 ml. of the diluted emulsion to the cement in the dish and stir the mixture at once with the steel rod, using a circular motion, making 60 complete revolutions during 1 min. Immediately at the end of the 1-min. mixing period, add 150 ml. of distilled water and continue the stirring for 3 min. The ingredients and apparatus shall be maintained at a temperature of approximately 25 C. during the mixing period.

(*d*) Pour the mixture through the tared 1410-micron (No. 14) iron sieve, of approximately 3 in. diameter and rinse by pouring distilled water from a receptacle held at a height of approximately 6 in. Place the sieve in a tared shallow pan, heat at 163 C. in an oven until dry, and weigh.

Report

32. Report the weight in grams of the material retained on the sieve and in the pan as the percentage of the emulsion broken.

COATING TEST

NOTE.—This test is applicable only for emulsions containing a base of asphalt of semisolid consistency. It is not applicable to the so-called quick-setting type of emulsions.

Apparatus and Material

33. The apparatus and material required shall consist of the following:

(*a*) *Screens.*—Standard $\frac{3}{4}$-in. and $\frac{1}{4}$-in. round-hole screens conforming to the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials.[3]

(*b*) *Spatula.*—A steel spatula or its equivalent, blade approximately 8 in. in length.

(*c*) *Dish.*—A round-bottom, iron dish or a kitchen saucepan, approximately 1-qt. capacity.

(*d*) *Stone.*—A supply of reference stone (hard limestone, trap rock, or other type) which has been washed with water and dried before using. The grading of this stone shall be such that it will all pass through a standard $\frac{3}{4}$-in. screen and not more than 5 per cent will pass through a $\frac{1}{4}$-in. screen.

NOTE.—Each laboratory shall select its own reference stone supply, the source of which is not apt to change. This is to obviate rapid changes in the character of reference stone used in any one laboratory.

Procedure

34. (*a*) Exactly 465 g. of the washed and dried graded stone, shall be weighed and placed in the metal pan.

(*b*) A 35-g. sample of the emulsion shall then be added to the stone in the pan and mixed vigorously with the spatula for 3 min.

(*c*) Record whether or not there is appreciable separation of the asphaltic base from the water of the emulsion and whether or not the stone is uniformly and thoroughly coated with the emulsion.

FREEZING TEST

Procedure

35. (*a*) Approximately 400 g. of the emulsion shall be placed in a clean metal container, such as a 1-pt. press-top tin.

(*b*) The emulsion in the closed container shall be exposed to a temperature of 0 F. (−17.8 C.) for twelve consecutive hours.

(*c*) At the expiration of the freezing period, the emulsion shall be permitted to thaw by exposure of the container to the temperature of the laboratory.

(*d*) After the first operation of freezing and thawing, the procedure shall be repeated twice, so that the emulsion will have been subjected to three cycles of freezing and thawing.

(*e*) After the third cycle, the emulsion may be homogeneous or may have separated into distinct layers which cannot be rendered homogeneous by stirring at laboratory temperature.

(*f*) The result of this test shall be reported as either "Homogeneous" or "Broken."

PENETRATION OF RESIDUE

36. The penetration shall be determined upon a representative portion of the residue in accordance with the Standard Method of Test for Penetration of Bituminous Materials (A.S.T.M. Designation: D 5) of the American Society for Testing Materials.[3]

SOLUBILITY OF RESIDUE

37. The solubility in carbon disulfide shall be determined upon a representative portion of the residue in accordance with the Standard Method of Test for Determination of Bitumen (A.S.T.M. Designation: D 4) of the American Society for Testing Materials.[3]

DUCTILITY OF RESIDUE

38. The ductility shall be determined upon a representative portion of the residue in accordance with the Standard Method of Test for Ductility of Bituminous Materials (A.S.T.M. Designation: D 113) of the American Society for Testing Materials.[3]

SPECIFIC GRAVITY OF RESIDUE

39. The specific gravity shall be determined upon a representative portion of the residue in accordance with the Standard Method of Test for Specific Gravity of Road Oils, Road Tars, Asphalt Cements, and Soft Tar Pitches (A.S.T.M. Designation: D 70) of the American Society for Testing Materials.[3]

ASH IN RESIDUE

40. The ash shall be determined upon a representative portion of the residue in accordance with the rapid routine method of ash determination, as described in Section 5 of the Standard Methods of Analysis of Grease (A.S.T.M. Designation: D 128) of the American Society for Testing Materials.[3]

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A.A.S.H.O. NO.: T 50-42

Standard Method of

FLOAT TEST FOR BITUMINOUS MATERIALS[1]

A.S.T.M. Designation: D 139 - 27

ADOPTED, 1927.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 139; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method covers the float test for bituminous materials.

Apparatus

2. (*a*) *Float.*—The float (Fig. 1) shall be made of aluminum or aluminum alloy and shall be in accordance with the following requirements:

	Min.	Normal	Max.
Weight of float, g...	37.70	37.90	38.10
Total height of float, mm.....	34.0	35.0	36.0
Height of rim above lower side of shoulder, mm.....	26.5	27.0	27.5
Thickness of shoulder, mm.....	1.3	1.4	1.5
Diameter of opening, mm.....	11.0	11.1	11.2

(*b*) *Collar.*—The collar (Fig. 1) shall be made of brass and shall be in accordance with the following requirements:

	Min.	Normal	Max.
Weight of collar, g...	9.60	9.80	10.00
Over-all height of collar, mm.....	22.3	22.5	22.7
Inside diameter at bottom, mm.....	12.72	12.82	12.92
Inside diameter at top, mm.....	9.65	9.70	9.75

The top of the collar shall screw up tightly against the lower side of the shoulder.

(*c*) *Calibration of Assembly.*—The assembled float and collar, with the collar filled flush with the bottom and weighted to a total weight of 53.2 g., shall float upon water with the rim 8.5 ± 1.5 mm. above the surface of the water. Dimensions of the apparatus additional to those required above are given in Fig. 1.

(*d*) *Thermometer.*—An A.S.T.M. Low Softening Point Thermometer, graduated in either Centigrade or Fahrenheit degrees as specified, having a range of −2 to +80 C. or +30 to +180 F. and conforming to the requirements for this thermometer as prescribed in the Standard Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1) of the American Society for Testing Materials.[3]

(*e*) *Bath.*—The diameter of the bath and the depth of water shall be at least 185 mm.

Preparation of Sample

3. (*a*) The brass collar shall be placed with the smaller end on a brass plate

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] Prior to adoption as standard, this method was published as tentative from 1922 to 1927, being revised in 1923, 1924, 1925, and 1927. Editorially revised in 1939.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

which has been previously amalgamated with mercury by first rubbing it with a dilute solution of mercuric chloride or nitrate, and then with mercury.

(*b*) The sample shall be completely melted at the lowest possible temperature that will bring it to a sufficiently fluid condition for pouring, excepting creosote-oil residues, which shall be mixed and poured at a temperature of 100 to 125 C. It shall be stirred thoroughly until it is homogeneous and free from air bubbles. The sample shall then be poured into the collar in any convenient manner until slightly more than level with the top.

(*c*) *Asphalt and Asphalt Products.*—Asphalt and asphalt products shall be cooled to room temperature, placed in water maintained at 5 C. for 5 min., after which the surplus material shall be removed by means of a spatula, or steel knife, which has been slightly heated. The collar and plate shall then be placed in a tin cup containing ice water maintained at 5 ± 1 C., and left in this bath for at least 15 min.

(*d*) *Tar Products.*—Tar products shall be immediately immersed in ice water maintained at 5 C. for 5 min., after which the surplus material shall be removed by means of a spatula or steel knife, which has been slightly heated. The collar and plate shall then be placed in a tin cup containing ice water maintained at 5 ± 1 C., and left in this bath for at least 15 min.

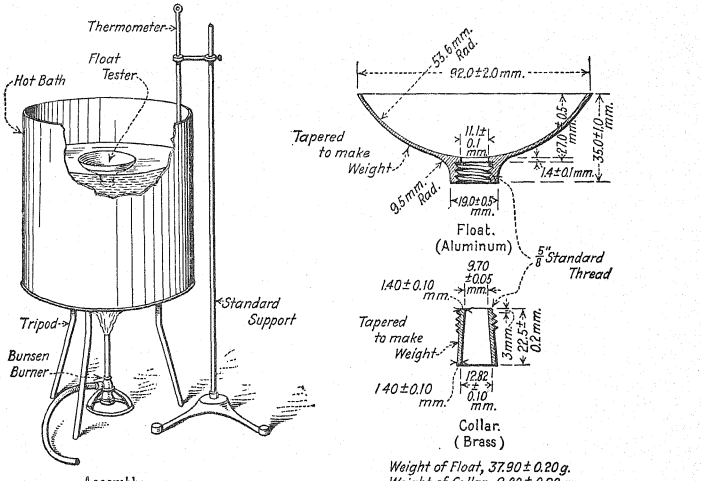


FIG. 1.—Float Test Apparatus.

Procedure

4. (*a*) The bath shall be filled with water and the water heated to the temperature at which the test is to be made. This temperature shall be accurately maintained and shall at no time throughout the test be allowed to vary more than 0.5 C. from the temperature specified.

(*b*) After the material to be tested has been kept in the ice water for not less than 15 min. nor more than 30 min., the collar with its contents shall be removed from the plate and screwed into the aluminum float and immersed in water at 5 C. for 1 min. Any water shall then be removed from the inside of the float and the latter immediately floated in the warm bath. As the plug of material becomes warm and fluid, it is forced upward and out of the collar until the water gains entrance into the saucer and causes it to sink.

(*c*) The time in seconds between placing the apparatus on the water and when the water breaks through the material shall be determined by means of a stop watch, and shall be taken as a measure of the consistency of the material under examination.

NOTE.—Special precaution should be taken to insure the collar fitting tightly into the float and to see that there is no seepage of water between the collar and float during the test.

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A.A.S.H.O. NO.: T 49-42

Standard Method of Test for

PENETRATION OF BITUMINOUS MATERIALS[1]

A.S.T.M. Designation: D 5 - 25

ADOPTED, 1911; REVISED, 1916, 1921, 1925.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 5; the final number indicates the year of original adoption as standard or in the case of revision, the year of last revision.

Scope

1. This method of test is intended for the determination of the penetration of bituminous materials.

Definition

2. Penetration is defined as the consistency of a bituminous material, expressed as the distance that a standard needle vertically penetrates a sample of the material under known conditions of loading, time, and temperature. Where the conditions of test are not specifically mentioned, the load, time, and temperature are understood to be 100 g., 5 sec., 25 C. (77 F.), respectively, and the units of penetration to indicate hundredths of a centimeter.

Apparatus

3. The apparatus shall consist of the following:

(*a*) *Container.*—A container, in which the sample is tested, made of metal or glass, cylindrical in shape, and having a flat bottom. Its inside dimensions shall be substantially as follows: 55 mm. (2.17 in.) in diameter, and 35 mm. (1.38 in.) in depth.

NOTE.—Containers known in the drug trade as seamless "ointment boxes" may be obtained in dimensions conforming to the above requirements.

(*b*) *Needle.*—A needle (Fig. 1) made from a cylindrical steel rod approximately 50.8 mm. (2 in.) in length, and 1.00 to 1.02 mm. in diameter. It shall be symmetrically tapered at one end to a cone approximately 6.35 mm. ($\frac{1}{4}$ in.) in height and whose angle shall be within the range of 8 deg. 40 min. and 9 deg. 40 min. After tapering, the point shall be "blunted" by grinding off to a truncated cone, the smaller base of which shall be from 0.14 to 0.16 mm. in diameter. The finished needle shall be hardened and highly polished.

NOTE.—The National Bureau of Standards will measure and certify the accuracy of penetration needles in accordance with the permissible variations given.

(*c*) *Water Bath.*—A water bath maintained at a temperature not varying more than 0.1 C. from 25 C. The volume of water shall be not less than 10 liters. The sample shall be immersed in the bath to a depth of not less than 10 cm.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.
[2] Editorially revised in 1939.

(4 in.) and shall be supported on a perforated shelf not less than 5 cm. (2 in.) from the bottom of the bath.

(*d*) *Penetration Apparatus.*—Any apparatus which will allow the needle to penetrate without appreciable friction, and which is accurately calibrated to yield results in accordance with the definition of penetration, will be acceptable.

(*e*) *Transfer Dish for Container.*—The transfer dish for container shall be a small dish or tray of such capacity as will insure complete immersion of the container during the test. It shall be provided with some means which will insure a firm bearing and prevent rocking of the container.

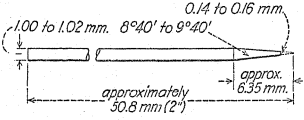


FIG. 1.—Needle for Penetration Test.

Preparation of Sample

4. The sample shall be completely melted at the lowest possible temperature and stirred thoroughly until it is homogeneous and free from air bubbles. It shall then be poured into the sample container to a depth of not less than 15 mm. ($\frac{5}{8}$ in.). The sample shall be protected from dust and allowed to cool in an atmosphere not lower than 18 C. (65 F.) for 1 hr. It shall then be placed in the water bath along with the transfer dish and allowed to remain 1 hr.

Procedure

5. (*a*) In making the test the sample shall be placed in the transfer dish filled with water from the water bath of sufficient depth to completely cover the container. The transfer dish containing the sample shall then be placed upon the stand of the penetration machine. The needle loaded with the specified weight, shall be adjusted to make contact with the surface of the sample. This may be accomplished by making contact of the actual needle point with its image reflected by the surface of the sample from a properly placed source of light. Either the reading of the dial shall then be noted or the pointer brought to zero. The needle shall then be released for the specified period of time, after which the penetration machine shall be adjusted to measure the distance penetrated.

(*b*) At least three tests shall be made at points on the surface of the sample not less than 1 cm. ($\frac{3}{8}$ in.) from the side of the container and not less than 1 cm. ($\frac{3}{8}$ in.) apart. After each test the sample and transfer dish shall be returned to the water bath and the needle shall be carefully wiped toward its point with a clean, dry cloth to remove all adhering bitumen. The reported penetration shall be the average of at least three tests whose values shall not differ more than four points between maximum and minimum.

(*c*) When desirable to vary the temperature, time and weight, and, in order to provide for a uniform method of reporting results when variations are made, the samples shall be melted and cooled in air as directed in Section 4. They shall then be immersed in water or brine, as the case may require, for 1 hr. at the temperature desired. The following combinations are suggested:

At 0 C. (32 F.)	200-g. weight, 60 sec.
At 46.1 C. (115F.)	50-g. weight, 5 sec.

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A.A.S.H.O. NO.: T 56-42

Standard Method of Test for

RESIDUE OF SPECIFIED PENETRATION[1]

A.S.T.M. Designation: D 243 – 36

ADOPTED, 1936.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 243; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test is intended for the determination of percentage of residue having a specified penetration at 100 g., 5 sec., 25 C. (77 F.), obtained by heating a road oil or a semisolid asphalt having a penetration of more than 100, at a temperature of 249 to 260 C. (480 to 500 F.). When the penetration of the residue is not otherwise stated it shall be understood to be 100. The residue obtained is available for tests as desired.

Apparatus

2. The apparatus shall consist of a container, heating bath, hot plate, and thermometer, with necessary accessory apparatus as follows:

(*a*) *Container.*—The container in which the sample is to be tested shall be a flat-bottom, cylindrical seamless tin box, 70 mm. ($2\frac{3}{4}$ in. in diameter and 45 mm. ($1\frac{3}{4}$ in.) in depth.

NOTE.—Containers known in the drug trade as seamless "ointment boxes" may be obtained in dimensions conforming to the above requirements.

(*b*) *Heating Bath.*—The heating bath shall be a cast-iron air bath permitting the immersion of the container to a depth of $1\frac{1}{4}$ in. through an opening $\frac{1}{16}$ in. larger in diameter than the container. It shall support the container $\frac{1}{4}$ in. above the hot plate and with at least $\frac{1}{4}$ in. free air space between the sides of the container and of the air bath below the opening. A suitable air bath is shown in Fig. 1.

(*c*) *Hot Plate.*—The air bath shall be heated upon a suitably mounted hot plate, heated either electrically or by means of a gas flame. The plate shall be capable of maintaining the sample continuously at the required temperature, and apparatus necessary to fulfill this requirement, such as a rheostat or gas pressure regulator, shall be provided.

(*d*) *Thermometer.*—An A.S.T.M. Open Flash Thermometer, graduated in either Centigrade or Fahrenheit degrees as specified, having a range of −6 to +400 C. or +20 to +760 F. and conforming to the requirements for this thermometer as prescribed in the Standard Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1) of the American Society for Testing Materials.[3]

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] Prior to adoption as standard, this method was published as tentative from 1926 to 1936, being revised in 1928, 1932, and 1935. Editorially revised in 1939.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Preparation of Sample

3. The sample as received shall be thoroughly stirred and agitated, to insure a complete mixture before the portion for testing is removed.

Procedure

4. (*a*) A 100.00 ± 0.10-g. sample of the material to be tested shall be weighed into a tared container, which shall then be placed in the air bath in position to be heated. The thermometer shall be supported in the sample equidistant from the sides of the container and with the bottom of the bulb neither more than ¼ in. above nor touching the bottom of the container. The bulb shall be completely immersed in the sample throughout the heating. An assembly of the apparatus is shown in Fig. 2.

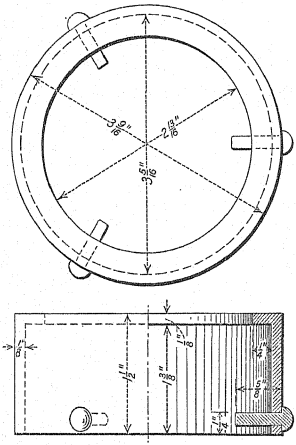


FIG. 1.—Cast-Iron Air Bath.

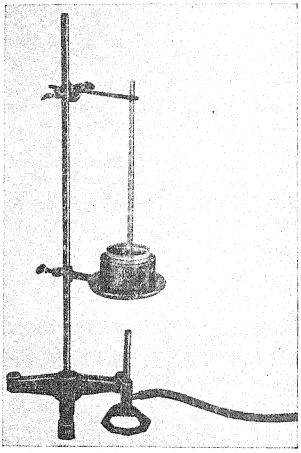


FIG. 2.—Assembly of Apparatus.

(*b*) The sample shall be heated as rapidly as possible without foaming to a temperature of 249 C. (480 F.) and during the evaporation, the temperature shall be maintained between 249 C. (480 F.) and 260 C. (500 F.). The sample shall be stirred with the thermometer from time to time to prevent local overheating and, to maintain a homogeneous sample, all cakes of hardened bitumen which form at the sides of the container shall be fluxed in the sample.

(*c*) An experienced operator can judge approximately what percentage of residue he should obtain to secure the desired penetration. When it is supposed that the residue will show the required penetration, the bitumen on the thermometer which may be readily scraped off shall be returned to the container, which then shall be removed from the air bath, cooled, and weighed. The penetration of the residue shall then be determined in accordance with the

Standard Method of Test for Penetration of Bituminous Materials (A.S.T.M. Designation: D 5) of the American Society for Testing Materials,[3] with the exception that the 6-oz. container specified in Section 2 (*a*), in which the evaporation has been conducted, shall be used instead of the 3-oz. container specified in the Standard Method D 5.

(*d*) It frequently is necessary to make several trials before a residue of the required penetration is obtained. If it is determined to be greater than that required, all water shall be removed from the container and the surface of the sample, and the heating and determination of penetration may be repeated as before. Ordinarily, a residue shall be considered as satisfactorily obtained when its penetration is within 15 of that desired, and its percentage by weight of the original sample shall be calculated. When it is necessary to determine more precisely the percentage of residue having the specified penetration, such a percentage shall be calculated by interpolation between percentages of two residues, one having a penetration greater and one having a penetration lower than that specified. The percentage shall be reported as:

Percentage of residue of - - - - - penetration (determined - - - - - -) stating, first, the specified penetration, and second, the penetration actually determined for the sample tested or calculated by interpolation.

Precautions

5. Certain types of road oil will readily form rings of hard asphalt at the side of the container. Great care should be taken that this material be completely fluxed in the sample before the penetration of the residue is determined.

Reproducibility of Results

6. With care and proper attention to details, duplicate determinations by this method should not differ from each other by more than 1.0 per cent with the same operator nor more than 2.5 per cent between different laboratories.

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BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: A37.10-1943
AMERICAN ASSOCIATION STATE
HIGHWAY OFFICIALS STANDARD
A.A.S.H.O. NO.: T 53-42

Standard Method of Test for

SOFTENING POINT OF BITUMINOUS MATERIALS[1]

RING-AND-BALL METHOD

A.S.T.M. Designation: D 36 - 26

ADOPTED, 1919; REVISED, 1921, 1924, 1926.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 36; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Softening Point

1. The softening of bituminous materials generally takes place at no definite moment or temperature. As the temperature rises, they gradually and imperceptibly change from a brittle or exceedingly thick and slow flowing material to a softer and less viscous liquid. For this reason the determination of the softening point must be made by a fixed, arbitrary, and closely defined method if the results obtained are to be comparable.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Ring.*—A brass ring 15.875 mm. ($\frac{5}{8}$ in.) in inside diameter and 6.35 mm. ($\frac{1}{4}$ in.) in depth; thickness of wall, 2.38 mm. ($\frac{3}{32}$ in.); permissible variation on inside diameter and thickness of ring 0.25 mm. (0.01 in.). This ring shall be attached in a convenient manner to a No. 13 B. & S. gage brass wire (diameter 1.83 mm. = 0.072 in.). See Fig. 1.

(*b*) *Ball.*—A steel ball 9.53 mm. ($\frac{3}{8}$ in.) in diameter weighing between 3.45 and 3.55 g.

(*c*) *Container.*—A glass vessel, capable of being heated, not less than 8.5 cm. (3.34 in.) in diameter and measuring 10.5 cm. (4.13 in.) in depth from the bottom of the flare. (A 600-ml. beaker, low form, meets this requirement.)

(*d*) *Thermometer.*—An A.S.T.M. Low Softening Point Thermometer, graduated in either Centigrade or Fahrenheit degrees as specified, having a range of −2 to +80 C. or +30 to +180 F. and conforming to the requirements for this thermometer as prescribed in the Standard Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1) of the American Society for Testing Materials.[3]

Preparation of Sample

3. The sample shall be melted and stirred thoroughly, avoiding incorporating air bubbles in the mass, and then poured into the ring so as to leave an

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] Prior to adoption as standard, this method was published as tentative from 1916 to 1919, being revised in 1919. Editorially revised in 1939 and 1942.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

excess on cooling. The ring, while being filled, should rest on a brass plate which has been amalgamated to prevent the bituminous material from adhering to it. After cooling 1 hr., the excess material shall be cut off cleanly with a slightly heated knife.

the sample in the water so that the lower surface of the filled ring is exactly 2.54 cm. (1 in.) above the bottom of the glass vessel and its upper surface is 5.08 cm. (2 in.) below the surface of the water. Place the ball in the water but not on the specimen. Suspend the

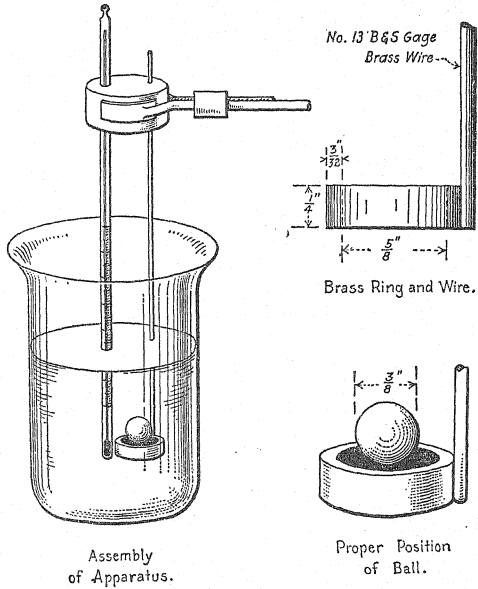


FIG. 1.—Apparatus for Ring-and-Ball Method.

Procedure for Bituminous Materials Having Softening Points 80C. (176 F.) or Below

Assembly of Apparatus

4. Fill the glass vessel to a depth of substantially 8.25 cm. (3.25 in.) with freshly boiled, distilled water at 5 C. (41 F.). Suspend the ring containing thermometer so that the bottom of the bulb is level with the bottom of the ring and within 0.635 cm. (¼ in.), but not touching the ring. Maintain the temperature of the water at 5 C. (41 F.) for 15 min. With suitable forceps, place the ball in the center of the upper surface of the bitumen in the ring, thus completing the assembly as in Fig. 1.

Heating

5. Apply the heat in such a manner that the temperature of the water is raised 5 C. (9 F.) each minute.

Softening Point

6. The temperature recorded by the thermometer at the instant the bituminous material touches the bottom of the glass vessel shall be reported as the softening point. No correction shall be made for emergent stem of the thermometer.

Permissible Variation in Rise of Temperature

7. The rate of rise of temperature shall be uniform and shall not be averaged over the period of the test. The maximum permissible variation for any minute period after the first three shall be plus or minus 0.5 C. (0.9 F.). All tests in which the rate of rise in temperature exceeds these limits shall be rejected.

Procedure for Bituminous Materials Having Softening Points Above 80 C. (176 F.)

Thermometer

8. An A.S.T.M. High Softening Point Thermometer, graduated in either Centigrade or Fahrenheit degrees as specified, having a range of 30 to 200 C. or 85 to 392 F. and conforming to the requirements for this thermometer as prescribed in the Standard Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1) of the American Society for Testing Materials[3] shall be used.

Modifications for Hard Materials

9. The same procedure as described in Sections 4 to 7 shall be employed except that U. S. P. glycerin shall be used instead of water, and the starting point of the glycerin bath shall be 32 C. (89.6 F.). The bath shall be brought to this temperature and thoroughly agitated, then the apparatus and specimens shall be placed in the bath which shall be maintained under agitation at the starting temperature for 15 min., after which the assembly shall be completed by placing the ball on the center of the specimen and the test carried on in accordance with Sections 4 to 7. In applying the heat, the ring apparatus shall be placed off the center of the container and the burner placed midway between the center and edge of the beaker away from the specimen.

Precautions

10. (*a*) The use of freshly boiled distilled water is essential, as otherwise air bubbles may form on the specimen and affect the accuracy of the results. Rigid adherence to the prescribed rate of heating is absolutely essential in order to secure accuracy of results.

(*b*) A sheet of paper placed on the bottom of the glass vessel and conveniently weighted will prevent the bituminous material from sticking to the glass vessel, thereby saving considerable time and trouble in cleaning.

Accuracy

11. The limit of accuracy of the test is plus or minus 0.5 C. (0.9 F.).

Standard Method of Test for

SOFTENING POINT OF TAR PRODUCTS[1]

CUBE-IN-WATER METHOD

A.S.T.M. Designation: D 61 – 38

ADOPTED, 1920; REVISED, 1924, 1938.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 61; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. (*a*) The softening of pitch takes place at no definite moment or temperature. As the temperature rises, pitch gradually and imperceptibly changes from a brittle or exceedingly thick and slow flowing material to a softer and less viscous liquid. For this reason the determination of the softening point must be made by a fixed, arbitrary, and closely defined method if the results obtained are to be comparable.

(*b*) The methods of test herein described are not applicable to pitches having softening points above 80 C. (176 F.).

(*c*) For the purpose of shortening the time required for testing, hard pitches having softening points between 43 and 80 C. (109.4 and 176 F.) are cooled at 15.5 C. (60 F.) instead of at 4 C. (39.2 F.) as prescribed for soft pitches.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Mold.*—A mold suitable for forming a 12.7-mm. ($\frac{1}{2}$-in.) cube of pitch. (Suitable types are shown in Fig. 1.)

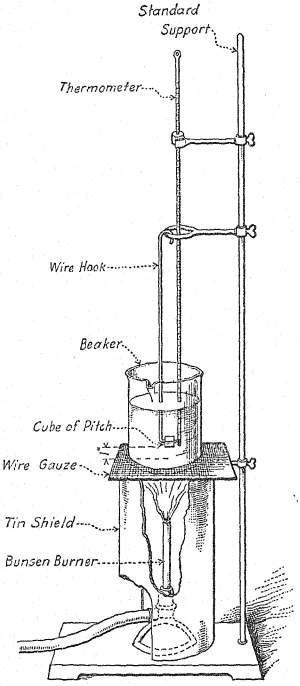
(*b*) *Hook.*—An L-shaped right-angled hook made of No. 12 B. & S. gage copper wire (diameter 2.05 mm. (0.0808 in.)) the foot of which shall be 2.54 cm. (1 in.) in length. See Fig. 1.

(*c*) *Container.*—A glass vessel, capable of being heated, not less than 8.5 cm. (3.34 in.) in diameter and measuring 10.5 cm. (4.13 in.) in depth from the bottom of the flare. (A 600-ml. beaker, Griffin low form, meets this requirement.)

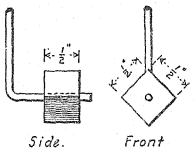
(*d*) *Thermometer.*—An A.S.T.M. Low Softening Point Thermometer, graduated in either Centigrade or Fahrenheit degrees as specified, having a range of −2 to +80 C. or +30 to +180 F. and conforming to the requirements for this thermometer as prescribed in the Standard Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation:

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

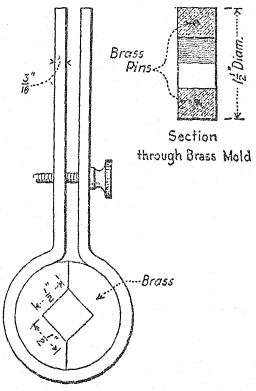
[2] Prior to adoption as standard, this method was published as tentative from 1919 to 1920. Editorially revised in 1942.



Assembly

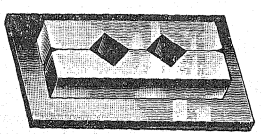


Method of Placing Cube on Wire Hook.

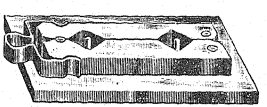


Section through Brass Mold

W. I. Pitch Mold (Special)



Double Mold



Double Mold with Pins

FIG. 1.—Apparatus for Cube-in-Water Method.

E 1) of the American Society for Testing Materials.[3]

Preparation of Sample

3. The pitch shall be formed into a 12.7-mm. ($\frac{1}{2}$-in.) cube, truly shaped and with sharp edges, either by melting and pouring, or softening and pressing, into a mold. In all cases an excess of pitch shall be used and the surplus material shall be cut off cleanly with a slightly heated knife. The harder pitches specified can ordinarily be molded at room temperature, the softer pitches in water at about 4 C. (39.2 F.). If they are melted, they shall first be thoroughly stirred, avoiding incorporating air bubbles in the mass, and then poured into the mold so as to leave an excess on cooling. The mold shall rest on a brass plate and the surface of the plate and the interior surfaces of the mold should be amalgamated to prevent the pitch from adhering to them.

A. Pitches Having Softening Points Between 43 and 80 C. (109.4 and 176 F.)

Procedure

4. (*a*) The apparatus shall be assembled as shown in Fig. 1. The glass vessel shall be filled to a depth of substantially 9.5 cm. (3.75 in.) with freshly distilled water (Note 1) at 15.5 C. (60 F.). The cube of pitch shall be placed on the wire as shown in Fig. 1 and suspended in the water so that its lower edge is exactly 2.54 cm. (1 in.) above the bottom of the glass vessel and its upper edge is 5.08 cm. (2 in.) below the surface of the water. The cube shall be allowed to remain in the water for 15 min. before the heat is applied. The thermometer shall be suspended so that the bottom of the bulb is level with the bottom edge of the cube of pitch and within 0.635 cm. ($\frac{1}{4}$ in.), but not touching the cube.

(*b*) The heat shall be applied in such a manner that the temperature of the water is raised 5 C. (9 F.) each minute. The rate of rise of temperature shall be uniform and shall not be averaged over the period of the test. The maximum permissible variation for any minute period after the first three shall be plus or minus 0.5 C. (0.9 F.). All tests in which the rate of rise in temperature exceeds these limits shall be rejected. Rigid adherence to the prescribed rate of heating is absolutely essential in order to secure accuracy of results.

(*c*) *Softening Point.*—The temperature recorded by the thermometer at the instant the pitch touches the bottom of the glass vessel (Note 2) shall be reported as the softening point. No correction shall be made for emergent stem of the thermometer.

NOTE 1.—The use of freshly distilled water is essential, as otherwise air bubbles may form on the cube and retard its sinking.

NOTE 2.—A sheet of paper placed on the bottom of the glass vessel and conveniently weighted will prevent the pitch from sticking to the glass vessel, thereby saving considerable time and trouble in cleaning.

B. Pitches Having Softening Points Below 43 C. (109.4 F.)

Procedure

5. For testing pitches having softening points below 43 C. (109.4 F.) the same procedure described under method *A* in Section 4 shall be followed, except that the water when placed in the glass vessel shall be at a temperature of 4 C. (39.2 F.). The cube shall be allowed to remain 15 min. in this water before the heat is applied.

Reproducibility of Results

6. With care and proper attention to details, duplicate determinations of softening point by this method should not differ from each other by more than 1.0 C. (1.8 F.).

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Method of Test for

SPECIFIC GRAVITY OF ASPHALTS AND TAR PITCHES SUFFICIENTLY SOLID TO BE HANDLED IN FRAGMENTS[1]



A.S.T.M. Designation: D 71 - 27

ADOPTED, 1927.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 71; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test is intended for the determination of the specific gravity of asphalts and tar pitches sufficiently solid to be handled in fragments.

Specific Gravity

2. The specific gravity of asphalts and tar pitches shall be expressed as the ratio of the weight of a given volume of the material at 25 C. (77 F.) to that of an equal volume of water at the same temperature and shall be expressed thus:

Specific gravity, 25/25 C. (77/77 F.)-----.

Apparatus

3. The determination of specific gravity shall be made with an analytical balance equipped with a pan straddle or other stationary support (Fig. 1).

Test Specimen

4. The test specimen shall be a cube of the material measuring approximately ½ in. to the edge. It shall be prepared by melting a small sample of the material by the gentle application of heat, care being exercised to prevent loss by evaporation, and pouring when sufficiently fluid into a ½-in. brass cubical mold, which has been amalgamated with mercury and which is placed on an amalgamated brass plate. Precautions should be taken to prevent the inclusion of air bubbles. The hot material should slightly more than fill the mold and when cool the excess may be cut off with a hot spatula. The specimen shall be removed from the mold when cooled to room temperature.

Procedure

5. The balance shall first be tared with a piece of fine waxed silk thread sufficiently long to reach from the hook on one of the pan supports to the straddle or rest. The test specimen shall then be attached to the thread, so as to be suspended about 1 in. above the straddle from the hook on the pan support, and weighed to the nearest 0.1 mg. This

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] Prior to adoption as standard, this method was published as tentative from 1920 to 1927.

weight is called "*a*." The specimen, still suspended by the thread, shall then be weighed, to the nearest 0.1 mg., completely immersed in freshly boiled distilled water at 25 ± 1 C. (77 ± 1.8 F.), adhering air bubbles being first removed with a fine wire. This weight is called "*b*."

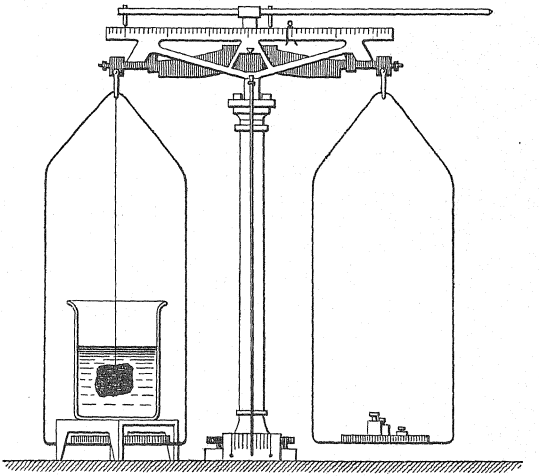


FIG. 1.—Analytical Balance Equipped with Pan Straddle.

Calculations

6. The specific gravity of the material shall be calculated from the following formula:

$$\text{Sp. gr.} = \frac{a}{a - b}$$

Accuracy

7. The limit of accuracy of the test is plus or minus 0.005 specific gravity.

Standard Method of Test for

SPECIFIC GRAVITY OF ROAD OILS, ROAD TARS, ASPHALT CEMENTS, AND SOFT TAR PITCHES[1]

A.S.T.M. Designation: D 70 - 27

ADOPTED, 1927.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 70; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test is intended for the determination of the specific gravity of road oils, road tars, asphalt cements, and soft tar pitches.

Specific Gravity

2. The specific gravity of road oils, road tars, asphalt cements, and soft tar pitches shall be expressed as the ratio of the weight of a given volume of the material at 25 C. (77 F.) to that of an equal volume of water at the same temperature and shall be expressed thus:

Specific gravity, 25/25 C. (77/77 F.)----

Apparatus

3. The determination of specific gravity shall be made with a pycnometer or weighing bottle (Fig. 1), which shall consist of a straight-walled glass tube approximately 70 mm. in length and 22 mm. in diameter, carefully ground to receive an accurately fitting solid glass stopper with a hole of 1.5 to 1.7-mm. bore in place of the usual capillary opening. The lower part of the stopper shall be made concave in order to allow all air bubbles to escape through the bore. The depth of the cup-shape depression shall be about 4.8 mm. at the center. The stoppered tube shall have a capacity of about 24 ml. and when empty shall weigh not over 35 g.

Weighing Pycnometer

4. Before making a determination, the pycnometer with stopper shall first be calibrated by weighing it clean and dry upon an analytical balance. This weight is called "*a*." It shall then be filled with freshly boiled distilled water at a temperature of 25 C. (77 F.), the stopper firmly inserted, all surplus moisture wiped from the surface with a clean dry cloth and again weighed. This weight is called "*b*."

Procedure for Road Oils and Tars

5. When determining the specific gravity of road oils or road tars which flow readily, the material shall be brought to a temperature of 25 C. (77 F.) and poured into the pycnometer until it is full, with care to prevent the inclusion of air bubbles. The stopper shall then be firmly inserted and all excess of

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] Prior to adoption as standard, this method was published as tentative from 1920 to 1927.

material forced through the opening and carefully removed with a clean dry cloth. The pycnometer and contents shall then be weighed. This weight is called "*c*." The specific gravity of the material shall be calculated from the formula:

$$\text{Sp. gr.} = \frac{c - a}{b - a}$$

Procedure for Asphalt Cements and Pitches

6. When determining the specific gravity of tar and asphalt products which are too viscous for the method described in Section 4, a small amount of the material shall be brought to a fluid condition by the gentle application of heat, care being exercised to prevent loss

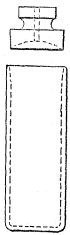


FIG. 1.—Pycnometer or Weighing Bottle.

by evaporation. When sufficiently fluid, enough of the sample shall be poured into the clean dry pycnometer to about half fill it. Precautions shall be taken to keep the material from touching the sides of the tube above the final level and to prevent the inclusion of air bubbles. The tube should be slightly warmed before filling. The pycnometer and contents shall then be cooled to room temperature and weighed with the stopper. This weight is called "*c*." The pycnometer shall next be removed from the balance, filled with freshly boiled distilled water, and the stopper firmly inserted. It shall then be completely immersed for not less than 30 min. in a beaker of distilled water maintained at 25 C. (77 F.), after which it shall be removed and all surplus water wiped off with a clean cloth. The pycnometer and contents shall then be weighed immediately. This weight is called "*d*." The specific gravity of the material shall be calculated from the formula:

$$\text{Sp. gr.} = \frac{c - a}{(b - a) - (d - c)}$$

Precautions

7. When making the specific gravity determination it is important that:

(*a*) Only freshly boiled distilled water shall be used.

(*b*) When weighing the pycnometer completely filled, the temperature of its contents shall be within 1 C. (1.8 F.) of 25 C. (77 F.).

(*c*) Precautions shall be taken to prevent expansion and overflow of the contents from the heat of the hand when wiping the surface of the pycnometer.

(*d*) The presence of all air bubbles shall be eliminated in filling the pycnometer and inserting the stopper.

(*e*) Weighings shall be made quickly after filling the pycnometer and shall be accurate to 1 mg. A number of trial fillings and catch weights may be necessary to obtain the desired degree of accuracy.

(*f*) To prevent breakage of the pycnometer when cleaning it out after a determination has been made upon a very viscous or semisolid material, it will be found advisable to warm it in an oven at not over 100 C. (212 F.) until most of the material may be poured out and then to swab it with a piece of soft cloth or cotton waste. When cool it may be finally rinsed with carbon disulfide, benzol, or other solvent and wiped clean.

Accuracy

8. The limit of accuracy of the test is plus or minus 0.005 specific gravity.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: Z11.2-1944

AMERICAN PETROLEUM INSTITUTE
STANDARD. A.P.I. NO.: 518-44

AMERICAN ASSOCIATION STATE
HIGHWAY OFFICIALS STANDARD
A.A.S.H.O. NO.: T 72-44

Standard Method of Test for
VISCOSITY BY MEANS OF THE SAYBOLT VISCOSIMETER[1]

A.S.T.M. Designation: D 88 – 44

ADOPTED, 1926; REVISED, 1930, 1933, 1936, 1938, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 88; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. (*a*) This method of test shall be used for determining the Saybolt viscosity[3] of petroleum products and lubricants. of flow of more than 32 sec. There is no maximum limit to viscosity to be measured by the Saybolt Universal Viscosimeter but, in general, liquids having

TABLE I.—DIMENSIONS OF OIL TUBES.

Dimensions	Saybolt Universal Viscosimeter			Saybolt Furol Viscosimeter		
	Minimum, cm.	Normal, cm.	Maximum, cm.	Minimum, cm.	Normal, cm.	Maximum, cm.
Inside diameter of outlet tube	0.1750	0.1765	0.1780	0.313	0.315	0.317
Outside diameter of outlet tube at lower end	0.28	0.30	0.32	0.40	0.43	0.46
Length of outlet tube[a]	1.215	1.225	1.235	1.215	1.225	1.235
Height of overflow rim above bottom of outlet tube[a]	12.40	12.50	12.60	12.40	12.50	12.60
Outside diameter of overflow rim, at the top[a, c]	[b]		3.30	[b]		3.30
Diameter of container[a]	2.955	2.975	2.995	2.955	2.975	2.995
Depth of cylindrical part of container[a]	8.8	.....	.....	8.8	....	....
Diameter of container between bottom of cylindrical part of container and top of outlet tube[a]	0.9	.....	.....	0.9	....	....

[a] This dimension is identical in the Saybolt Universal and the Saybolt Furol instruments.
[b] The minimum value shall preferably be not less than 3.2 cm.
[c] The section of overflow rim shall be bounded by straight lines, except that a fillet is permissible at the junction with the bottom of the gallery.

(*b*) The Saybolt Universal Viscosimeter shall be used only for oils with times an outflow time of the order of 1000 sec. and higher, Saybolt Universal, are tested more conveniently by means of the Saybolt Furol Viscosimeter.

(*c*) The Saybolt Furol Viscosimeter shall be used only for oils with times of flow of more than 25 sec. The outflow time of the Furol (Note) instrument is approximately one-tenth that of the Universal.

NOTE.—The word "Furol" is a contraction of the phrase "fuel and road oils."

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants.

[2] Prior to adoption as standard, this method was published as tentative from 1921 to 1926, being revised in 1923 when it was combined with the Methods of Test for Viscosity by Means of the Saybolt Standard Universal Viscosimeter (D 47—21), and again revised in 1924, 1925, and 1926.

[3] The Conversion of Saybolt Universal Viscosity and Saybolt Furol Viscosity to Kinematic Viscosities in centistokes is covered by the Standard Method for Conversion of Kinematic Viscosity to Saybolt Universal Viscosity (A.S.T.M. Designation: D 446) and Standard Method for Conversion of Kinematic Viscosity to Saybolt Furol Viscosity (A.S.T.M. Designation: D 666), which methods appear in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Apparatus

2. The apparatus shall consist of an oil tube, bath, receiver, thermometers, timer, and withdrawal tube, conforming to the requirements specified in the following Paragraphs (*a*) to (*g*):

(*a*) *Oil Tube.*—The oil tube (illustrated in Fig. 1) shall be entirely of corrosion-resistant metal, with or without plating, and shall conform to the dimensional requirements shown in Table I

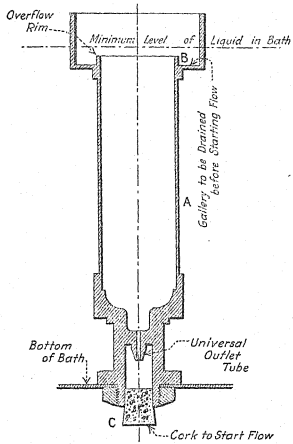


FIG. 1.—Sectional View of Standard Oil Tube.

within the permissible variations prescribed. The lower end of the oil tube shall be provided with a nut for locking it in place in the bath and with a cork or other suitable device to prevent flow until the test is started. For convenience a string or its equivalent may be attached to the cork for rapid removal.

The oil tube shall be standardized, and any correction in excess of 0.2 per cent shall be applied. The time of flow shall be within plus or minus 1 per cent (Note 1) of the time as obtained with the National Bureau of Standards' master Saybolt oil tube (Note 2).

NOTE 1.—For routine testing, oil tubes having corrections as high as plus or minus 2 per cent may be employed, provided, however, that any new oil tubes obtained after the adoption of this method shall not have corrections higher than plus or minus 1 per cent. For referee testing, oil tubes having corrections higher than plus or minus 1 per cent shall not be employed.

The oil tube may be standardized by any one of the following methods:

(*a*) Determination of outflow time for American Petroleum Institute oil standard and application of appropriate correction to obtain established value for the oil standard.

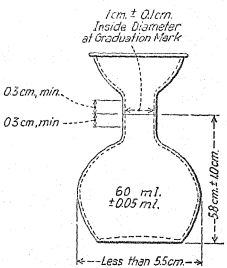


FIG. 2.—Receiving Flask.
(This type of flask is recommended as convenient and durable.)

(*b*) Calibration by National Bureau of Standards subsequent to January 1, 1935.

(*c*) Comparison with a tube certified by the National Bureau of Standards subsequent to January 1, 1935, provided the tube shows no evidence of damage or injury since certification.

NOTE 2.—This tube, conforming to the A.S.T.M. dimensional specifications, has outflow times calibrated against oil standards under the maintenance of the American Petroleum Institute Committee on Viscosity Standards.

(*b*) *Bath.*—The bath shall serve as a support to hold the oil tube in a vertical

position and as a container for the bath liquid. The bath shall be equipped with a stirring device and with means for heating or cooling. The source of heat or refrigeration shall be more than 1¼ in. (3 cm.) from the oil tube. If an external heater is used it shall be more than 2 in. (5 cm.) from the oil tube.

The bath temperature necessary to maintain thermal equilibrium (while the oil in the oil tube is well stirred by the oil-tube thermometer) shall be within plus or minus 0.1 F. (0.06 C.) of the standard temperatures of 70 F. (21.1 C.) or 77 F. (25 C.) or shall not exceed 100.25, 122.35, 130.5, 141.0, 181.5 or 212.0 F. (37.9, 50.2, 54.7, 60.6, 83.1 or 100.0 C.), respectively, for the standard temperatures mentioned in Section 3. The level of the bath liquid shall not be lower than ¼ in. (0.5 cm.) above the overflow rim of the oil tube. For referee testing the bath liquid must be one which in the bath used will meet the preceding bath-temperature conditions (Note).

NOTE.—These temperature requirements can be met with water, aqueous solutions, and some baths with oil. In routine testing oil is generally used as the bath medium. This is allowable provided the temperature of the oil bath is adjusted so that the necessary condition of thermal equilibrium is maintained. It may be necessary to maintain the oil bath at slightly higher temperatures than those specified in the preceding paragraph. Temperature differentials between oil bath and oil tube necessary to maintain thermal equilibrium may be double those given above.

(*c*) *Receiver.*—The receiving flask (see Fig. 2) shall be of glass with a capacity up to the graduation mark on its neck of 60 ± 0.05 ml. at 68 F. (20 C.). At the graduation mark, the inside diameter of the neck of the flask shall be 1 ± 0.1 cm. The cylindrical portion of the neck of the flask shall extend not less than 0.3 cm. above and below the graduation mark. The graduation mark shall be 5.8 ± 1.0 cm. above the bottom of the flask. The maximum outside diameter shall be less than 5.5 cm.

NOTE.—Many years' experience with flasks conforming to these dimensions has proved it to be satisfactory for all but the most viscous oils. With the latter, drainage errors may occur if the length of the neck of the flask above the graduation mark varies excessively. As soon as stocks of flasks now on hand have been depleted, it is planned to revise the flask's specification by limiting the length of the neck above the graduation mark to a maximum of 11 mm.

(*d*) *Oil-Tube Thermometers.*—The A.S.T.M. Saybolt Viscosity Thermometers shall include two sets of six thermometers each, one set being graduated in Fahrenheit degrees and the other in Centigrade degrees, the ranges being chosen to include the temperatures commonly used in testing as indicated in the following table. They shall conform to the requirements for these thermometers as prescribed in the Standard Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1) of the American Society for Testing Materials.[4] To prevent contact of the thermometer with the orifice in the oil tube a suitable support shall be attached to the enlargement of the thermometer stem.

Temperature Range	Subdivisions	Temperature of Tests
FAHRENHEIT THERMOMETERS		
66 to 80 F.	0.2 F.	70 and 77 F.
94 to 108 F.	0.2 F.	100 F.
120 to 134 F.	0.2 F.	122 and 130 F.
134 to 148 F.	0.2 F.	140 F.
174 to 188 F.	0.2 F.	180 F.
204 to 218 F.	0.2 F.	210 F.
CENTIGRADE THERMOMETERS		
19 to 27 C.	0.1 C.	21.1 and 25 C.
34 to 42 C.	0.1 C.	37.8 C.
49 to 57 C.	0.1 C.	50 and 54.4 C.
57 to 65 C.	0.1 C.	60 C.
79 to 87 C.	0.1 C.	82.2 C.
95 to 103 C.	0.1 C.	98.9 C.

(*e*) *Bath Thermometers.*—Thermometers as specified in Paragraph (*d*) or

[4] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

other means for measuring temperature of at least equal accuracy, shall be used in the bath.

(*f*) *Timer.*—The stop watch or other timing device used shall be graduated in divisions of 0.2 sec. or less, and shall be accurate to within 0.1 per cent when tested over a 60-min. period (Note).

NOTE.—Electrical timing devices are permissible provided they are accurate and capable of being read to 0.2 sec. Timing devices actuated by synchronous motors shall be used only on electric circuits of controlled frequency.

(*g*) *Withdrawal Tube.*—The tube or pipette used for draining the gallery shall have a smooth tip of about 3 mm. outside diameter and about 2 mm. inside diameter.

Temperature of Testing

3. (*a*) With the Saybolt Universal Viscosimeter, determinations shall be made at 70, 100, 130 or 210 F. (21.1, 37.8, 54.4 or 98.9 C.).

(*b*) With the Saybolt Furol Viscosimeter, determinations shall be made at 77. 100, 122 or 210 F. (25, 37.8, 50 or 98.9 C.). In tests on road and paving materials, determinations may also be made at 140 and 180 F. (60 and 82.2 C.).

Procedure

4. (*a*) Viscosity determinations shall be made in a room free from drafts and rapid changes in temperature. For standardization and referee tests the room shall be between 68 and 86 F (20 and 30 C.) and the actual temperature shall be recorded. For routine tests, temperatures up to 100 F. may be employed without introducing errors in excess of 1 per cent. Determinations shall not be made at temperatures below the dew point of the atmosphere surrounding the instrument.

(*b*) The oil tube shall first be cleaned with an effective solvent, such as benzol, and excess solvent shall be removed from the gallery.

(*c*) All oil shall be passed through a 100-mesh wire strainer before it is introduced into the oil tube. After the tube is cleaned, a quantity of the oil to be tested, sufficient to wet the entire surface of the tube, shall be poured into the tube and allowed to drain out (Note). The cork stopper shall be inserted not less than $\frac{1}{4}$ in. nor more than $\frac{3}{8}$ in. into the lower end of the air chamber at the bottom of the oil tube. The cork shall fit tightly enough to prevent the escape of air, as evidenced by the absence of oil on the cork after it is withdrawn.

NOTE.—The plunger commonly supplied with the viscosimeter shall never be used on instruments maintained as standards. In case the flash point is less than 50 F. (27.8 C.) above the temperature of test, the wetting operation should be omitted in order to avoid an increase in viscosity due to evaporation of a portion of the light fraction.

(*d*) If the test temperature is above that of the room, the oil shall be heated to not more than 3 F. (1.7 C.) above the temperature of test and if the temperature is below that of the room, the oil shall be cooled to not more than 3 F. (1.7 C.) below the temperature of test. In no case, however, shall the oil be preheated to a temperature above 50 F. (27.8 C.) below the flash point. The oil shall be poured into the oil tube until it ceases to overflow into the gallery. The oil in the oil tube shall be kept well stirred with the oil-tube thermometer, care being taken to avoid hitting the outflow tube. The bath temperature shall be adjusted until the oil temperature remains constant. After thermal equilibrium has been attained no further adjustments shall be made in the bath temperature. The test results shall be discarded if the indicated bath temperature varies by more than plus or minus 0.05 F. (0.03 C.) in tests at 70, 77, 100,

122 and 130 F. (21.1, 25, 37.8, 50 and 54.4 C.) or by more than plus or minus 0.10 F. (0.06 C.) in tests at 140, 180 and 210 F. (60, 82.2 and 98.9 C.).

(*e*) After the temperature of the oil in the oil tube has remained constant within 0.02 F. (0.01 C.) of the desired temperature for 1 min. with constant stirring, the oil-tube thermometer shall be withdrawn and the surplus oil removed quickly from the gallery by means of the withdrawal tube so that the level of the oil in the gallery is below the level in the oil tube proper. The tip of the withdrawal tube shall be inserted at one point in the gallery. The test shall be started over again if the tip of the withdrawal tube touches the overflow rim (see Fig. 1). Under no conditions shall the excess oil be removed by rotating the withdrawal tube around the gallery.

(*f*) The receiving flask shall be placed in position so that the stream of oil from the outlet tube will strike the neck of the flask. The graduation mark on the receiving flask shall be not less than 10 cm. nor more than 13 cm. from the bottom of the bath. The cork shall be snapped from its position and at the same instant the timer shall be started. The timer shall be stopped when the bottom of the meniscus of the oil reaches the mark on the neck of the receiving flask.

Reporting Results

5. The time in seconds as determined by the prescribed procedure, with the proper calibration correction, is the Saybolt Universal (or Saybolt Furol) viscosity of the oil at the temperature at which the test is made. Results shall be reported to the nearest 0.1 sec. for viscosity values below 200 sec. and to the nearest whole second for values 200 sec. or above.

Reproducibility of Results

6. With proper attention to details of method of procedure, results in different laboratories with different operators under referee or standardization conditions of testing, should not differ by more than 0.5 per cent.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: Z11.53-1944

AMERICAN PETROLEUM INSTITUTE
STANDARD. A.P.I. NO.: 548-44

Standard Method for

CONVERSION OF KINEMATIC VISCOSITY TO SAYBOLT FUROL VISCOSITY[1]

A.S.T.M. Designation: D 666 - 44

ADOPTED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 666; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. The conversion table in this method provides a means for converting kinematic viscosity in centistokes at 122 F. to Saybolt Furol viscosity in seconds at 122 F.[3]

Procedure

2. For the kinematic viscosities at 122 F. listed in Table I the equivalent Saybolt Furol viscosity in seconds at 122 F. may be read directly. For kinematic viscosities between 48 and 400 centistokes which are not listed in Table I, the equivalent Saybolt Furol viscosity values may be obtained by linear interpolation. For kinematic viscosities over 400 centistokes, the equivalent Saybolt Furol viscosity may be calculated by use of the multiplication constant given in Table I.

TABLE I.—VALUES FOR CONVERTING KINEMATIC VISCOSITY TO SAYBOLT FUROL VISCOSITY AT 122 F.

Kinematic Viscosity, centistokes	Saybolt Furol Viscosity, sec.	Kinematic Viscosity, centistokes	Saybolt Furol Viscosity, sec.	Kinematic Viscosity, centistokes	Saybolt Furol Viscosity, sec.
48	25.2	100	48.5	200	94.7
50	26.0	105	50.8	210	99.3
52	26.9	110	53.1	220	104.0
54	27.8	115	55.3	230	108.7
56	28.7	120	57.6	240	113.3
58	29.6	125	59.9	250	118.0
60	30.5	130	62.2	260	122.7
62	31.4	135	64.5	270	127.4
64	32.3	140	66.9	280	132.0
66	33.2	145	69.2	290	136.7
68	34.1	150	71.5	300	141.3
70	35.0	155	73.8	310	146.0
72	35.9	160	76.1	320	150.7
74	36.8	165	78.4	330	155.4
76	37.7	170	80.7	340	160.1
78	38.6	175	83.1	350	164.8
80	39.5	180	85.4	360	169.5
82	40.4	185	87.7	370	174.1
84	41.3	190	90.0	380	178.8
86	42.2	195	92.4	390	183.5
88	43.1			400	188.2
90	44.0				
92	44.9				
94	45.8				
96	46.7				
98	47.6				

Kinematic Viscosity, centistokes	Saybolt Furol Viscosity, sec.
Over 400	Saybolt seconds = centistokes × 0.470

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants.

[2] Prior to adoption as standard, this method was published as tentative from 1942 to 1944.

[3] The test procedure for determining kinematic viscosity is described in the Tentative Method of Test for Kinematic Viscosity (A.S.T.M. Designation: D 445); the test procedure for determining Saybolt Furol Viscosity appears in the Standard Method of Test for Viscosity by Means of the Saybolt Viscosimeter (A.S.T.M. Designation: D 88), which methods appear in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
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AMERICAN PETROLEUM INSTITUTE
STANDARD. A.P.I. NO.: 500-36

STANDARD ABRIDGED VOLUME CORRECTION TABLE FOR PETROLEUM OILS[1]

A.S.T.M. Designation: D 206 – 36

ADOPTED, 1925; REVISED, 1934, 1936.

This Standard of the American Society for Testing Materials is issued under the fixed designation D 206; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

This abridged table has been prepared by the National Bureau of Standards to meet a demand from the oil industry for a short and convenient table for reducing oil volumes to the basis of 60 F., when extreme accuracy is not required. It is not intended to replace the Bureau of Standards *Circular C 410*, but rather to supplement it and especially to replace the various abridged tables heretofore employed in the oil industry.

The groups, coefficients of expansion, gravity (degrees A.P.I.), and gravity ranges for the several subdivisions of the present abridged table are as follows:

Group Number	Coefficient of Expansion per deg. Fahr. at 60 F	Gravity, Degrees A.P.I.	Gravity Range of Group (Degrees A.P.I. at 60 F.)
0.....	0.00035	6	Up to 14.9
1.....	0.00040	22	15.0 to 34.9
2.....	0.00050	44	35.0 to 50.9
3.....	0.00060	58	51.0 to 63.9
4.....	0.00070	72	64.0 to 78.9
5.....	0.00080	86	79.0 to 88.9
6.....	0.00085	91	89.0 to 93.9
7.....	0.00090	97	94.0 to 100.0

All motor fuel blends of gasoline and benzol shall be considered as falling in Group 3. In cases of uncertainty regarding the presence of benzol, a product having a gravity heavier (numerically less) than 51° A.P.I., and a 50 per cent recovery point less than 293 F. (145 C.) shall be considered as falling in Group 3.

This table shows the volume occupied at 60 F. by a quantity of oil occupying unit volume at the indicated temperature.

For purposes of brevity the columns showing observed temperatures in degrees Fahrenheit carry the heading "t." The columns of multipliers which are actually $\left(\frac{\text{volume at 60 F.}}{\text{volume at "t"}}\right)$ carry the heading "M."

NOTE: *Example.*—The values given in the table are in the form of multipliers; that is, the volume of oil at the indicated temperature and degrees A.P.I. for each group, multiplied by the corresponding factor in the table, equals the volume at 60 F. For example, if the degrees A.P.I. of an oil at 60 F. equals 58 (Group 3) and the volume at 88 F. equals 8000 gal., then the volume at 60 F. equals 8000 × 0.9830 or 7864 gal.

[1] Under the standardization procedure of the Society, this standard is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants.

This abridged table differs from that published in 1934 in three respects: The multipliers for Group 0 have been revised slightly; Group 1 has been extended from 249 to 499 F.; and Groups 4, 5 and 6 have been extended from 99 to 124 F.

The multipliers in the Group 0 table in the 1934 standard were taken from the National Bureau of Standards *Miscellaneous Publication No. 97.* With the revision of *Circular No. 154* (published as *Circular C 410*) to include data on oils from 0 to 10° A.P.I., it has seemed desirable to employ the procedure followed in the other groups and choose the column of multipliers from the unabridged table which has a base coefficient of expansion nearest 0.00035, which in the case of Group 0 is the column corresponding to a gravity of 6° A.P.I.

GROUP 0[a]

Legend: t = observed temperature in degrees Fahrenheit; M = multiplier for reducing oil volumes to the basis of 60 F.

t	M	t	M	t	M	t	M	t	M	t	M	t	M
0....	1.0211	65..	0.9982	130..	0.9758	195..	0.9539	260..	0.9328	325..	0.9123	390..	0.8923	455..	0.8729
1....	1.0208	66..	0.9979	131..	0.9755	196..	0.9536	261..	0.9325	326..	0.9119	391..	0.8920	456	0.8726
2....	1.0204	67..	0.9976	132..	0.9751	197..	0.9533	262..	0.9322	327..	0.9116	392..	0.8917	457..	0.8723
3....	1.0201	68..	0.9972	133..	0.9748	198..	0.9530	263..	0.9319	328..	0.9113	393..	0.8914	458..	0.8720
4....	1.0197	69..	0.9969	134..	0.9745	199..	0.9527	264..	0.9315	329..	0.9110	394..	0.8911	459..	0.8717
5....	1.0194	70..	0.9965	135..	0.9741	200..	0.9523	265..	0.9312	330..	0.9107	395..	0.8908	460..	0.8714
6....	1.0190	71..	0.9962	136..	0.9738	201..	0.9520	266..	0.9309	331..	0.9104	396..	0.8905	461..	0.8711
7....	1.0186	72..	0.9958	137..	0.9735	202..	0.9517	267..	0.9306	332..	0.9101	397..	0.8902	462..	0.8709
8....	1.0183	73..	0.9955	138..	0.9731	203..	0.9513	268..	0.9303	333..	0.9098	398..	0.8899	463..	0.8706
9....	1.0179	74..	0.9952	139..	0.9728	204..	0.9510	269..	0.9299	334..	0.9095	399..	0.8896	464..	0.8703
10....	1.0176	75..	0.9948	140..	0.9724	205..	0.9507	270..	0.9296	335..	0.9092	400..	0.8893	465..	0.8700
11....	1.0172	76..	0.9944	141..	0.9721	206..	0.9504	271..	0.9293	336..	0.9088	401..	0.8890	466..	0.8697
12....	1.0168	77..	0.9941	142..	0.9718	207..	0.9500	272..	0.9290	337..	0.9085	402..	0.8887	467..	0.8694
13....	1.0165	78..	0.9938	143..	0.9714	208..	0.9497	273..	0.9287	338..	0.9082	403..	0.8884	468..	0.8691
14....	1.0161	79..	0.9934	144..	0.9711	209..	0.9494	274..	0.9283	339..	0.9079	404..	0.8881	469..	0.8688
15....	1.0158	80..	0.9931	145..	0.9707	210..	0.9490	275..	0.9280	340..	0.9076	405..	0.8878	470..	0.8685
16....	1.0154	81..	0.9927	146..	0.9704	211..	0.9487	276..	0.9277	341..	0.9073	406..	0.8875	471..	0.8682
17....	1.0151	82..	0.9924	147..	0.9701	212..	0.9484	277..	0.9274	342..	0.9070	407..	0.8872	472..	0.8679
18....	1.0147	83..	0.9920	148..	0.9697	213..	0.9481	278..	0.9271	343..	0.9067	408..	0.8869	473..	0.8676
19....	1.0144	84..	0.9917	149..	0.9694	214..	0.9477	279..	0.9267	344..	0.9064	409..	0.8866	474..	0.8673
20....	1.0141	85..	0.9914	150..	0.9691	215..	0.9474	280..	0.9264	345..	0.9061	410..	0.8863	475..	0.8671
21....	1.0137	86..	0.9910	151..	0.9687	216..	0.9471	281..	0.9261	346..	0.9057	411..	0.8860	476..	0.8668
22....	1.0134	87..	0.9907	152..	0.9684	217..	0.9468	282..	0.9258	347..	0.9054	412..	0.8857	477..	0.8665
23....	1.0130	88..	0.9903	153..	0.9680	218..	0.9464	283..	0.9255	348..	0.9051	413..	0.8854	478..	0.8662
24....	1.0126	89..	0.9900	154..	0.9677	219..	0.9461	284..	0.9252	349..	0.9048	414..	0.8851	479..	0.8659
25....	1.0123	90..	0.9896	155..	0.9674	220..	0.9458	285..	0.9248	350..	0.9045	415..	0.8848	480..	0.8656
26....	1.0119	91..	0.9892	156..	0.9670	221..	0.9454	286..	0.9245	351..	0.9042	416..	0.8845	481..	0.8653
27....	1.0116	92..	0.9889	157..	0.9667	222..	0.9451	287..	0.9242	352..	0.9039	417..	0.8842	482..	0.8650
28....	1.0112	93..	0.9886	158..	0.9664	223..	0.9448	288..	0.9239	353..	0.9036	418..	0.8839	483..	0.8647
29....	1.0109	94..	0.9882	159..	0.9660	224..	0.9445	289..	0.9236	354..	0.9033	419..	0.8836	484..	0.8644
30....	1.0106	95..	0.9879	160..	0.9657	225..	0.9441	290..	0.9233	355..	0.9030	420..	0.8833	485..	0.8642
31....	1.0102	96..	0.9876	161..	0.9654	226..	0.9438	291..	0.9229	356..	0.9027	421..	0.8830	486..	0.8639
32....	1.0098	97..	0.9872	162..	0.9650	227..	0.9435	292..	0.9226	357..	0.9024	422..	0.8827	487..	0.8636
33....	1.0095	98..	0.9869	163..	0.9647	228..	0.9432	293..	0.9223	358..	0.9021	423..	0.8824	488..	0.8633
34....	1.0092	99..	0.9865	164..	0.9643	229..	0.9428	294..	0.9220	359..	0.9017	424..	0.8821	489..	0.8630
35....	1.0088	100..	0.9862	165..	0.9640	230..	0.9425	295..	0.9217	360..	0.9014	425..	0.8818	490..	0.8627
36....	1.0084	101..	0.9858	166..	0.9637	231..	0.9422	296..	0.9214	361..	0.9011	426..	0.8815	491..	0.8624
37....	1.0081	102..	0.9855	167..	0.9633	232..	0.9419	297..	0.9210	362..	0.9008	427..	0.8812	492..	0.8621
38....	1.0077	103..	0.9852	168..	0.9630	233..	0.9415	298..	0.9207	363..	0.9005	428..	0.8809	493..	0.8618
39....	1.0074	104..	0.9848	169..	0.9627	234..	0.9412	299..	0.9204	364..	0.9002	429..	0.8806	494..	0.8615
40....	1.0070	105..	0.9844	170..	0.9623	235..	0.9409	300..	0.9201	365..	0.8999	430..	0.8803	495..	0.8613
41....	1.0067	106..	0.9841	171..	0.9620	236..	0.9406	301..	0.9198	366..	0.8996	431..	0.8800	496..	0.8610
42....	1.0063	107..	0.9837	172..	0.9616	237..	0.9402	302..	0.9195	367..	0.8993	432..	0.8797	497..	0.8607
43....	1.0059	108..	0.9834	173..	0.9613	238..	0.9399	303..	0.9191	368..	0.8990	433..	0.8794	498..	0.8604
44....	1.0056	109..	0.9831	174..	0.9610	239..	0.9396	304..	0.9188	369..	0.8987	434..	0.8791	499..	0.8601
45....	1.0052	110..	0.9827	175..	0.9606	240..	0.9392	305..	0.9185	370..	0.8984	435..	0.8788		.....
46....	1.0049	111..	0.9823	176..	0.9603	241..	0.9389	306..	0.9182	371..	0.8981	436..	0.8785		.....
47....	1.0045	112..	0.9820	177..	0.9600	242..	0.9386	307..	0.9179	372..	0.8978	437..	0.8782		.....
48....	1.0042	113..	0.9816	178..	0.9596	243..	0.9383	308..	0.9176	373..	0.8975	438..	0.8779		.....
49....	1.0039	114..	0.9813	179..	0.9593	244..	0.9380	309..	0.9173	374..	0.8972	439..	0.8776		.....
50....	1.0035	115..	0.9809	180..	0.9590	245..	0.9376	310..	0.9169	375..	0.8969	440..	0.8773		.....
51....	1.0032	116..	0.9806	181..	0.9586	246..	0.9373	311..	0.9166	376..	0.8965	441..	0.8770		.....
52....	1.0028	117..	0.9802	182..	0.9583	247..	0.9370	312..	0.9163	377..	0.8962	442..	0.8767		.....
53....	1.0025	118..	0.9799	183..	0.9580	248..	0.9367	313..	0.9160	378..	0.8959	443..	0.8764		.....
54....	1.0021	119..	0.9795	184..	0.9576	249..	0.9364	314..	0.9157	379..	0.8956	444..	0.8762		.....
55....	1.0017	120..	0.9792	185..	0.9573	250..	0.9360	315..	0.9154	380..	0.8953	445..	0.8759		.....
56....	1.0014	121..	0.9789	186..	0.9569	251..	0.9357	316..	0.9151	381..	0.8950	446..	0.8756		.....
57....	1.0010	122..	0.9785	187..	0.9566	252..	0.9354	317..	0.9148	382..	0.8947	447..	0.8753		.....
58....	1.0007	123..	0.9782	188..	0.9563	253..	0.9351	318..	0.9145	383..	0.8944	448..	0.8750		.....
59....	1.0003	124..	0.9779	189..	0.9559	254..	0.9347	319..	0.9141	384..	0.8941	449..	0.8747		.....
60....	1.0000	125..	0.9775	190..	0.9556	255..	0.9344	320..	0.9138	385..	0.8938	450..	0.8744		.....
61....	0.9997	126..	0.9772	191..	0.9553	256..	0.9341	321..	0.9135	386..	0.8935	451..	0.8741		.....
62....	0.9993	127..	0.9768	192..	0.9549	257..	0.9338	322..	0.9132	387..	0.8932	452..	0.8738		.....
63....	0.9990	128..	0.9765	193..	0.9546	258..	0.9335	323..	0.9129	388..	0.8929	453..	0.8735		.....
64....	0.9986	129..	0.9762	194..	0.9543	259..	0.9331	324..	0.9126	389..	0.8926	454..	0.8732		.....

[a] These tables for Group 0 and Group I have been adopted as standard by the Society as volume correction tables for asphaltic products on the joint recommendation of A.S.T.M. Committee D-4 on Road and Paving Materials and Committee D-8 on Bituminous Waterproofing and Roofing Materials.

STANDARD ABRIDGED VOLUME CORRECTION TABLE FOR PETROLEUM OILS

GROUP 1[a]

Legend: t = observed temperature in degrees Fahrenheit; M = multiplier for reducing oil volumes to the basis of 60 F.

t	M	t	M	t	M	t	M	t	M	t	M	t	M
0....	1.0242	65..	0.9980	130 .	0.9724	195..	0.9476	260..	0.9234	325..	0.8998	390..	0.8769	455..	0.8546
1....	1.0238	66..	0.9976	131..	0.9720	196..	0.9472	261..	0.9230	326..	0.8994	391..	0.8766	456..	0.8543
2....	1.0234	67..	0.9972	132..	0.9716	197..	0.9468	262..	0.9226	327..	0.8991	392..	0.8762	457..	0.8540
3....	1.0230	68..	0.9968	133..	0.9713	198..	0.9465	263..	0.9223	328..	0.8987	393..	0.8759	458..	0.8536
4....	1.0226	69..	0.9964	134..	0.9709	199..	0.9461	264..	0.9219	329..	0.8984	394..	0.8755	459..	0.8533
5....	1.0222	70..	0.9960	135 .	0.9705	200..	0.9457	265..	0.9215	330..	0.8980	395..	0.8752	460..	0.8530
6....	1.0218	71..	0.9956	136..	0.9701	201..	0.9453	266..	0.9212	331..	0.8977	396..	0.8748	461..	0.8526
7....	1.0214	72..	0.9952	137..	0.9697	202..	0.9449	267..	0.9208	332..	0.8973	397..	0.8745	462..	0.8523
8....	1.0210	73..	0.9948	138..	0.9694	203..	0.9446	268..	0.9204	333..	0.8969	398..	0.8741	463..	0.8519
9....	1.0206	74..	0.9944	139..	0.9690	204..	0.9442	269..	0.9201	334..	0.8966	399..	0.8738	464..	0.8516
10....	1.0202	75..	0.9940	140..	0.9686	205..	0.9438	270..	0.9197	335..	0.8962	400..	0.8734	465..	0.8513
11....	1.0198	76..	0.9936	141..	0.9682	206..	0.9434	271..	0.9193	336..	0.8959	401..	0.8731	466..	0.8509
12....	1.0194	77..	0.9932	142..	0.9678	207..	0.9430	272..	0.9190	337..	0.8955	402..	0.8727	467..	0.8506
13....	1.0189	78..	0.9929	143..	0.9675	208..	0.9427	273..	0.9186	338..	0.8952	403..	0.8724	468..	0.8503
14....	1.0185	79..	0.9925	144..	0.9671	209..	0.9423	274..	0.9182	339..	0.8948	404..	0.8720	469..	0.8499
15....	1.0181	80..	0.9921	145..	0.9667	210..	0.9419	275..	0.9179	340..	0.8945	405..	0.8717	470..	0.8496
16....	1.0177	81..	0.9917	146..	0.9663	211..	0.9415	276..	0.9175	341..	0.8941	406..	0.8714	471..	0.8493
17....	1.0173	82..	0.9913	147..	0.9659	212..	0.9412	277..	0.9171	342..	0.8938	407..	0.8710	472..	0.8489
18....	1.0169	83..	0.9909	148..	0.9655	213..	0.9408	278..	0.9168	343..	0.8934	408..	0.8707	473..	0.8486
19....	1.0165	84..	0.9905	149..	0.9651	214..	0.9405	279..	0.9164	344..	0.8930	409..	0.8703	474..	0.8482
20....	1.0161	85..	0.9901	150..	0.9647	215..	0.9401	280..	0.9160	345..	0.8927	410..	0.8700	475..	0.8479
21....	1.0157	86..	0.9897	151..	0.9643	216..	0.9397	281..	0.9157	346..	0.8923	411..	0.8696	476..	0.8476
22....	1.0153	87..	0.9893	152..	0.9639	217..	0.9393	282..	0.9153	347..	0.8920	412..	0.8693	477..	0.8472
23....	1.0148	88..	0.9889	153..	0.9636	218..	0.9390	283..	0.9149	348..	0.8916	413..	0.8689	478..	0.8469
24....	1.0144	89..	0.9885	154..	0.9632	219..	0.9386	284..	0.9146	349..	0.8913	414..	0.8686	479..	0.8466
25....	1.0140	90..	0.9881	155..	0.9628	220..	0.9382	285..	0.9142	350..	0.8909	415..	0.8683	480..	0.8462
26....	1.0136	91..	0.9877	156..	0.9624	221..	0.9378	286..	0.9139	351..	0.8906	416..	0.8679	481..	0.8459
27....	1.0132	92..	0.9873	157..	0.9620	222..	0.9374	287..	0.9135	352..	0.8902	417..	0.8676	482..	0.8456
28....	1.0128	93..	0.9869	158..	0.9616	223..	0.9371	288..	0.9131	353..	0.8899	418..	0.8672	483..	0.8452
29....	1 0124	94..	0.9865	159..	0.9612	224..	0.9367	289..	0.9128	354..	0.8895	419..	0.8669	484..	0.8449
30....	1.0120	95..	0.9861	160..	0.9608	225..	0.9363	290..	0.9124	355..	0.8892	420..	0.8665	485..	0.8446
31....	1.0116	96..	0.9857	161..	0.9604	226..	0.9359	291..	0.9120	356..	0.8888	421..	0.8662	486..	0.8442
32....	1.0112	97..	0.9853	162..	0.9601	227..	0.9356	292..	0.9117	357..	0.8884	422..	0.8659	487..	0.8439
33....	1.0108	98..	0.9849	163..	0.9597	228..	0.9352	293..	0.9113	358..	0.8881	423..	0.8655	488..	0.8436
34....	1.0104	99..	0.9845	164..	0.9594	229..	0.9349	294..	0.9110	359..	0.8877	424..	0.8652	489..	0.8432
35....	1.0100	100..	0.9841	165..	0.9590	230..	0.9345	295..	0.9106	360..	0.8874	425..	0.8648	490..	0.8429
36....	1.0096	101..	0.9837	166..	0.9586	231..	0.9341	296..	0.9102	361..	0.8870	426..	0.8645	491..	0.8426
37....	1.0092	102..	0.9833	167..	0.9582	232..	0.9337	297..	0.9099	362..	0.8867	427..	0.8641	492..	0.8422
38....	1.0088	103..	0.9830	168..	0.9578	233..	0.9334	298..	0.9095	363..	0.8863	428..	0.8638	493..	0.8419
39....	1.0084	104..	0.9826	169..	0.9574	234..	0.9330	299..	0.9091	364..	0.8860	429..	0.8635	494..	0.8416
40....	1.0080	105..	0.9822	170..	0.9570	235..	0.9326	300..	0.9088	365..	0.8856	430..	0.8631	495..	0.8413
41....	1.0076	106..	0.9818	171..	0.9566	236..	0.9322	301..	0.9084	366..	0.8853	431..	0.8628	496..	0.8409
42....	1.0072	107..	0.9814	172..	0.9562	237..	0.9318	302..	0.9081	367..	0.8849	432..	0.8624	497..	0.8406
43....	1.0068	108..	0.9811	173..	0.9559	238..	0.9315	303..	0.9077	368..	0.8846	433..	0.8621	498..	0.8403
44....	1.0064	109..	0.9807	174..	0.9555	239..	0.9311	304..	0.9073	369..	0.8842	434..	0.8618	499..	0.8399
45....	1.0060	110..	0.9803	175..	0.9551	240..	0.9307	305..	0.9070	370..	0.8839	435..	0.8614	.....	.....
46....	1.0056	111..	0.9799	176..	0.9547	241..	0.9303	306..	0.9066	371..	0.8835	436..	0.8611	.....	.....
47....	1.0052	112..	0.9795	177..	0.9543	242..	0.9300	307..	0.9063	372..	0.8832	437..	0.8607	.....	.....
48....	1.0048	113..	0.9791	178..	0.9540	243..	0.9296	308..	0.9059	373..	0.8828	438..	0.8604	.....	.....
49....	1.0044	114..	0.9787	179..	0.9536	244..	0.9293	309..	0.9055	374..	0.8825	439..	0.8601	.....	.....
50....	1.0040	115..	0.9783	180..	0.9532	245..	0.9289	310..	0.9052	375..	0.8821	440..	0.8597	.....	.....
51....	1.0036	116..	0.9779	181..	0.9528	246..	0.9285	311..	0.9048	376..	0.8818	441..	0.8594	.....	.....
52....	1.0032	117..	0.9775	182..	0.9524	247..	0.9281	312..	0.9045	377..	0.8814	442..	0.8590	.....	.....
53....	1.0028	118..	0.9771	183..	0.9521	248..	0.9278	313..	0.9041	378..	0.8811	443..	0.8587	.....	.....
54....	1.0024	119..	0.9767	184..	0.9517	249..	0.9274	314..	0.9037	379..	0.8807	444..	0.8584	.....	.....
55....	1.0020	120..	0.9763	185..	0.9513	250..	0.9270	315..	0.9034	380..	0.8804	445..	0.8580	.....	.....
56....	1.0016	121..	0.9759	186..	0.9509	251..	0.9267	316..	0.9030	381..	0.8800	446..	0.8577	.....	.....
57....	1.0012	122..	0.9755	187..	0.9505	252..	0.9263	317..	0.9027	382..	0.8797	447..	0.8573	.....	.....
58....	1.0008	123..	0.9752	188..	0.9502	253..	0.9259	318..	0.9023	383..	0.8793	448..	0.8570	.....	.....
59....	1.0004	124..	0.9748	189..	0.9498	254 .	0.9256	319..	0.9020	384..	0.8790	449..	0.8567	.....	.....
60....	1.0000	125..	0.9744	190..	0.9494	255..	0.9252	320..	0.9016	385..	0.8786	450..	0.8563	.....	.....
61....	0.9996	126..	0.9740	191..	0.9490	256..	0.9248	321..	0.9012	386..	0.8782	451..	0.8560	.....	.....
62....	0.9992	127..	0.9736	192..	0.9487	257..	0.9245	322..	0.9009	387..	0.8779	452..	0.8557	.....	.....
63....	0.9988	128..	0.9732	193..	0.9483	258..	0.9241	323..	0.9005	388..	0.8776	453..	0.8553	.....	.....
64....	0.9984	129..	0.9728	194..	0.9480	259..	0.9237	324..	0.9002	389..	0.8772	454..	0.8550	.....	.....

[a] These tables for Group 0 and Group 1 have been adopted as standard by the Society as volume correction tables for asphaltic products on the joint recommendation of A.S.T.M. Committee D-4 on Road and Paving Materials and Committee D-8 on Bituminous Waterproofing and Roofing Materials.

STANDARD ABRIDGED VOLUME CORRECTION TABLE FOR PETROLEUM OILS

Group 2

Legend: t = observed temperature in degrees Fahrenheit; M = multiplier for reducing oil volumes to the basis of 60 F.

t	M	t	M	t	M	t	M	t	M	t	M	t	M
0....	1.0297	20....	1.0198	40....	1.0099	60....	1.0000	80....	0.9901	100..	0.9802	120..	0.9702
1....	1.0292	21....	1.0193	41....	1.0094	61....	0.9995	81....	0.9896	101..	0.9797	121..	0.9697
2....	1.0287	22....	1.0188	42....	1.0089	62....	0.9990	82....	0.9891	102..	0.9792	122..	0.9692
3....	1.0283	23....	1.0184	43....	1.0084	63....	0.9985	83....	0.9886	103..	0.9786	123..	0.9688
4....	1.0278	24....	1.0179	44....	1.0079	64....	0.9980	84....	0.9881	104..	0.9781	124..	0.9683
5....	1.0273	25....	1.0174	45....	1.0074	65....	0.9975	85....	0.9876	105..	0.9776	125..	0.9677
6....	1.0268	26....	1.0169	46....	1.0069	66....	0.9970	86....	0.9871	106..	0.9771	126..	0.9672
7....	1.0263	27....	1.0164	47....	1.0064	67....	0.9965	87....	0.9866	107..	0.9766	127..	0.9667
8....	1.0258	28....	1.0159	48....	1.0059	68....	0.9961	88....	0.9861	108..	0.9762	128..	0.9662
9....	1.0253	29....	1.0154	49....	1.0054	69....	0.9956	89....	0.9856	109..	0.9757	129..	0.9657
10....	1.0248	30....	1.0149	50....	1.0049	70....	0.9951	90....	0.9851	110..	0.9752	130..	0.9652
11....	1.0243	31....	1.0144	51....	1.0044	71....	0.9946	91....	0.9846	111..	0.9747	131..	0.9647
12....	1.0238	32....	1.0139	52....	1.0039	72....	0.9941	92....	0.9841	112..	0.9742	132..	0.9642
13....	1.0233	33....	1.0134	53....	1.0035	73....	0.9935	93....	0.9836	113..	0.9737	133..	0.9637
14....	1.0228	34....	1.0129	54....	1.0030	74....	0.9930	94....	0.9831	114..	0.9732	134..	0.9632
15....	1.0223	35....	1.0124	55....	1.0025	75....	0.9925	95....	0.9826	115..	0.9727	135..	0.9627
16....	1.0218	36....	1.0119	56....	1.0020	76....	0.9920	96....	0.9821	116..	0.9722	136..	0.9622
17....	1.0213	37....	1.0114	57....	1.0015	77....	0.9915	97....	0.9816	117..	0.9717	137..	0.9617
18....	1.0208	38....	1.0109	58....	1.0010	78....	0.9911	98....	0.9812	118..	0.9712	138..	0.9613
19....	1.0203	39....	1.0104	59....	1.0005	79....	0.9906	99....	0.9807	119..	0.9707	139..	0.9608

Group 3

Legend: t = observed temperature in degrees Fahrenheit; M = multiplier for reducing oil volumes to the basis of 60 F.

t	M	t	M	t	M	t	M	t	M	t	M	t	M
0.....	1.0361	20.....	1.0241	40.....	1.0121	60.....	1.0000	80.....	0.9879	100....	0.9757	120....	0.9635
1.....	1.0355	21.....	1.0235	41.....	1.0115	61.....	0.9994	81.....	0.9873	101....	0.9751	121....	0.9629
2.....	1.0349	22.....	1.0229	42.....	1.0109	62.....	0.9988	82.....	0.9867	102....	0.9745	122....	0.9623
3.....	1.0343	23.....	1.0223	43.....	1.0103	63.....	0.9982	83.....	0.9860	103....	0.9738	123....	0.9617
4.....	1.0337	24.....	1.0217	44.....	1.0097	64.....	0.9976	84.....	0.9854	104....	0.9732	124....	0.9611
5.....	1.0331	25.....	1.0211	45.....	1.0091	65.....	0.9970	85.....	0.9848	105....	0.9726	.....	.....
6.....	1.0325	26.....	1.0205	46.....	1.0085	66.....	0.9964	86.....	0.9842	106....	0.9720	.....	.....
7.....	1.0319	27.....	1.0199	47.....	1.0079	67.....	0.9957	87.....	0.9836	107....	0.9714	.....	.....
8.....	1.0313	28.....	1.0193	48.....	1.0072	68.....	0.9951	88.....	0.9830	108....	0.9708	.....	.....
9.....	1.0307	29.....	1.0187	49.....	1.0066	69.....	0.9945	89.....	0.9824	109....	0.9702	.....	.....
10.....	1.0301	30.....	1.0181	50.....	1.0060	70.....	0.9939	90.....	0.9818	110....	0.9696	.....	.....
11.....	1.0295	31.....	1.0175	51.....	1.0054	71.....	0.9933	91.....	0.9812	111....	0.9690	.....	.....
12.....	1.0289	32.....	1.0169	52.....	1.0048	72.....	0.9927	92.....	0.9806	112....	0.9684	.....	.....
13.....	1.0283	33.....	1.0163	53.....	1.0042	73.....	0.9921	93.....	0.9800	113....	0.9678	.....	.....
14.....	1.0277	34.....	1.0157	54.....	1.0036	74.....	0.9915	94.....	0.9794	114....	0.9672	.....	.....
15.....	1.0271	35.....	1.0151	55.....	1.0030	75.....	0.9909	95.....	0.9788	115....	0.9666	.....	.....
16.....	1.0265	36.....	1.0145	56.....	1.0024	76.....	0.9903	96.....	0.9782	116....	0.9660	.....	.....
17.....	1.0259	37.....	1.0139	57.....	1.0018	77.....	0.9897	97.....	0.9776	117....	0.9654	.....	.....
18.....	1.0253	38.....	1.0133	58.....	1.0012	78.....	0.9891	98.....	0.9769	118....	0.9647	.....	.....
19.....	1.0247	39.....	1.0127	59.....	1.0006	79.....	0.9885	99.....	0.9763	119....	0.9641	.....	.....

Group 4

Legend: t = observed temperature in degrees Fahrenheit; M = multiplier for reducing oil volumes to the basis of 60 F.

t	M	t	M	t	M	t	M	t	M	t	M	t	M
0.....	1.0419	20.....	1.0280	40.....	1.0141	60.....	1.0000	80.....	0.9858	100....	0.9716	120....	0.9574
1.....	1.0412	21.....	1.0273	41.....	1.0134	61.....	0.9993	81.....	0.9851	101....	0.9709	121....	0.9567
2.....	1.0405	22.....	1.0266	42.....	1.0127	62.....	0.9986	82.....	0.9844	102....	0.9702	122....	0.9560
3.....	1.0398	23.....	1.0260	43.....	1.0120	63.....	0.9979	83.....	0.9837	103....	0.9695	123....	0.9552
4.....	1.0391	24.....	1.0253	44.....	1.0113	64.....	0.9972	84.....	0.9830	104....	0.9688	124....	0.9545
5.....	1.0384	25.....	1.0246	45.....	1.0106	65.....	0.9965	85.....	0.9823	105....	0.9681	.....	.....
6.....	1.0377	26.....	1.0239	46.....	1.0099	66.....	0.9958	86.....	0.9816	106....	0.9673	.....	.....
7.....	1.0370	27.....	1.0232	47.....	1.0092	67.....	0.9951	87.....	0.9809	107....	0.9666	.....	.....
8.....	1.0364	28.....	1.0225	48.....	1.0084	68.....	0.9943	88.....	0.9802	108....	0.9659	.....	.....
9.....	1.0357	29.....	1.0218	49.....	1.0077	69.....	0.9936	89.....	0.9795	109....	0.9652	.....	.....
10.....	1.0350	30.....	1.0211	50.....	1.0070	70.....	0.9929	90.....	0.9788	110....	0.9645	.....	.....
11.....	1.0343	31.....	1.0204	51.....	1.0063	71.....	0.9922	91.....	0.9780	111....	0.9638	.....	.....
12.....	1.0336	32.....	1.0197	52.....	1.0056	72.....	0.9915	92.....	0.9773	112....	0.9631	.....	.....
13.....	1.0329	33.....	1.0190	53.....	1.0049	73.....	0.9908	93.....	0.9766	113....	0.9624	.....	.....
14.....	1.0322	34.....	1.0183	54.....	1.0042	74.....	0.9901	94.....	0.9759	114....	0.9617	.....	.....
15.....	1.0315	35.....	1.0176	55.....	1.0035	75.....	0.9894	95.....	0.9752	115....	0.9609	.....	.....
16.....	1.0308	36.....	1.0169	56.....	1.0028	76.....	0.9887	96.....	0.9745	116....	0.9602	.....	.....
17.....	1.0301	37.....	1.0162	57.....	1.0021	77.....	0.9880	97.....	0.9738	117....	0.9595	.....	.....
18.....	1.0294	38.....	1.0155	58.....	1.0014	78.....	0.9872	98.....	0.9731	118....	0.9588	.....	.....
19.....	1.0287	39.....	1.0148	59.....	1.0007	79.....	0.9865	99.....	0.9723	119....	0.9581	.....	.....

Group 5

Legend: t = observed temperature in degrees Fahrenheit; M = multiplier for reducing oil volumes to the basis of 60 F.

t	M	t	M	t	M	t	M	t	M	t	M
0	1.0476	20	1.0319	40	1.0160	60	1.0000	80	0.9838	100	0.9676	120	0.9513
1	1.0468	21	1.0311	41	1.0152	61	0.9992	81	0.9830	101	0.9668	121	0.9505
2	1.0460	22	1.0303	42	1.0144	62	0.9984	82	0.9822	102	0.9660	122	0.9497
3	1.0453	23	1.0296	43	1.0136	63	0.9976	83	0.9814	103	0.9652	123	0.9489
4	1.0445	24	1.0288	44	1.0128	64	0.9968	84	0.9806	104	0.9643	124	0.9480
5	1.0437	25	1.0280	45	1.0120	65	0.9960	85	0.9798	105	0.9635		
6	1.0429	26	1.0272	46	1.0112	66	0.9952	86	0.9790	106	0.9627		
7	1.0421	27	1.0264	47	1.0104	67	0.9944	87	0.9782	107	0.9619		
8	1.0414	28	1.0256	48	1.0096	68	0.9935	88	0.9773	108	0.9611		
9	1.0406	29	1.0248	49	1.0088	69	0.9927	89	0.9765	109	0.9603		
10	1.0398	30	1.0240	50	1.0080	70	0.9919	90	0.9757	110	0.9594		
11	1.0390	31	1.0232	51	1.0072	71	0.9911	91	0.9749	111	0.9586		
12	1.0382	32	1.0224	52	1.0064	72	0.9903	92	0.9741	112	0.9578		
13	1.0375	33	1.0216	53	1.0056	73	0.9895	93	0.9733	113	0.9570		
14	1.0367	34	1.0208	54	1.0048	74	0.9887	94	0.9725	114	0.9562		
15	1.0359	35	1.0200	55	1.0040	75	0.9879	95	0.9717	115	0.9554		
16	1.0351	36	1.0192	56	1.0032	76	0.9871	96	0.9709	116	0.9546		
17	1.0343	37	1.0184	57	1.0024	77	0.9863	97	0.9700	117	0.9538		
18	1.0335	38	1.0176	58	1.0016	78	0.9854	98	0.9692	118	0.9529		
19	1.0327	39	1.0168	59	1.0008	79	0.9846	99	0.9684	119	0.9521		

Group 6

Legend: t = observed temperature in degrees Fahrenheit; M = multiplier for reducing oil volumes to the basis of 60 F.

t	M	t	M	t	M	t	M	t	M	t	M	t	M
0	1.0501	20	1.0355	40	1.0169	60	1.0000	80	0.9830	100	0.9659	120	0.9487
1	1.0493	21	1.0327	41	1.0160	61	0.9992	81	0.9821	101	0.9650	121	0.9479
2	1.0484	22	1.0319	42	1.0152	62	0.9983	82	0.9813	102	0.9642	122	0.9470
3	1.0476	23	1.0310	43	1.0143	63	0.9975	83	0.9804	103	0.9633	123	0.9462
4	1.0467	24	1.0302	44	1.0135	64	0.9966	84	0.9796	104	0.9625	124	0.9453
5	1.0459	25	1.0294	45	1.0126	65	0.9958	85	0.9787	105	0.9616		
6	1.0451	26	1.0286	46	1.0118	66	0.9949	86	0.9779	106	0.9608		
7	1.0443	27	1.0277	47	1.0110	67	0.9941	87	0.9770	107	0.9599		
8	1.0434	28	1.0269	48	1.0101	68	0.9932	88	0.9762	108	0.9590		
9	1.0426	29	1.0260	49	1.0093	69	0.9924	89	0.9753	109	0.9582		
10	1.0418	30	1.0252	50	1.0084	70	0.9915	90	0.9745	110	0.9573		
11	1.0410	31	1.0244	51	1.0076	71	0.9907	91	0.9736	111	0.9565		
12	1.0402	32	1.0236	52	1.0067	73	0.9898	92	0.9727	112	0.9556		
13	1.0393	33	1.0227	53	1.0059	73	0.9890	93	0.9719	113	0.9548		
14	1.0385	34	1.0219	54	1.0050	74	0.9881	94	0.9710	114	0.9539		
15	1.0377	35	1.0211	55	1.0042	75	0.9873	95	0.9702	115	0.9530		
16	1.0369	36	1.0203	56	1.0034	76	0.9864	96	0.9693	116	0.9522		
17	1.0360	37	1.0194	57	1.0025	77	0.9856	97	0.9685	117	0.9513		
18	1.0352	38	1.0186	58	1.0017	78	0.9847	98	0.9676	118	0.9505		
19	1.0343	39	1.0177	59	1.0008	79	0.9839	99	0.9668	119	0.9496		

Group 7

Legend: t = observed temperature in degrees Fahrenheit; M = multiplier for reducing oil volumes to the basis of 60 F.

t	M	t	M	t	M	t	M	t	M
0	1.0531	20	1.0356	40	1.0179	60	1.0000	80	0.9820
1	1.0522	21	1.0347	41	1.0170	61	0.9991	81	0.9811
2	1.0513	22	1.0338	42	1.0161	62	0.9982	82	0.9802
3	1.0505	23	1.0330	43	1.0152	63	0.9973	83	0.9792
4	1.0496	24	1.0321	44	1.0143	64	0.9964	84	0.9783
5	1.0487	25	1.0312	45	1.0134	65	0.9955	85	0.9774
6	1.0478	26	1.0303	46	1.0125	66	0.9946	86	0.9765
7	1.0470	27	1.0294	47	1.0116	67	0.9937	87	0.9756
8	1.0461	28	1.0286	48	1.0108	68	0.9928	88	0.9747
9	1.0453	29	1.0277	49	1.0099	69	0.9919	89	0.9738
10	1.0444	30	1.0268	50	1.0090	70	0.9910	90	0.9729
11	1.0435	31	1.0259	51	1.0081	71	0.9901	91	0.9719
12	1.0426	32	1.0250	52	1.0072	72	0.9892	92	0.9710
13	1.0418	33	1.0242	53	1.0063	73	0.9883	93	0.9701
14	1.0409	34	1.0233	54	1.0054	74	0.9874	94	0.9692
15	1.0400	35	1.0224	55	1.0045	75	0.9865	95	0.9683
16	1.0391	36	1.0215	56	1.0036	76	0.9856	96	0.9674
17	1.0382	37	1.0206	57	1.0027	77	0.9847	97	0.9665
18	1.0374	38	1.0197	58	1.0018	78	0.9838	98	0.9656
19	1.0365	39	1.0188	59	1.0009	79	0.9829	99	0.9647

APPROVED AS
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A.A.S.H.O. NO.: T 55-42

Standard Method of Test for

WATER IN PETROLEUM PRODUCTS AND OTHER BITUMINOUS MATERIALS[1]

A.S.T.M. Designation: D 95 – 40

ADOPTED, 1924; REVISED, 1927, 1928, 1930, 1940.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 95; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test is intended for use in the determination of the water in a sample of bituminous material by distilling the sample with a volatile solvent. The method is suitable for a variety of materials but is especially applicable to petroleum, fuel oil, road oil, coal tar, water-gas tar, coke-oven tar, and other petroleum products or bituminous materials.

NOTE.—For the determination of water in grease reference should be made to the procedure described in the Standard Methods of Analysis of Grease (A.S.T.M. Designation: D 128) of the American Society for Testing Materials.[3]

Apparatus

2. (*a*) The apparatus shall consist of a metal still or glass flask, heated by suitable means and provided with a reflux condenser discharging into a trap connected to the still or flask. The trap serves to collect and measure the condensed water and to return the solvent to the still. The type of distilling apparatus used is not an essential feature of this method, but glass has been generally used for petroleum products and the metal still for road materials and tars.

(*b*) *Metal Still.*—The metal still (Fig. 1 (*a*)) shall be a vertical cylindrical vessel, preferably of copper, having a faced flange at the top to which the head is tightly attached by means of a clamp. The head shall be of metal, preferably of brass or copper, and be provided with a tubulation 1 in. in inside diameter.

(*c*) *Glass Flask.*—The glass flask (Fig. 1 (*b*)) shall be of the short neck, round-bottom type, made of well-annealed glass, having an approximate capacity of 500 ml.

(*d*) *Heat Source.*—The burner used with the metal still shall be a ring gas burner 4 in. (100 mm.) in inside diameter. With the glass flask, an ordinary gas burner or electric heater may be used as the source of heat.

(*e*) *Condenser.*—The condenser shall be of the water-cooled, reflux, glass-tube type, having a condenser jacket not less

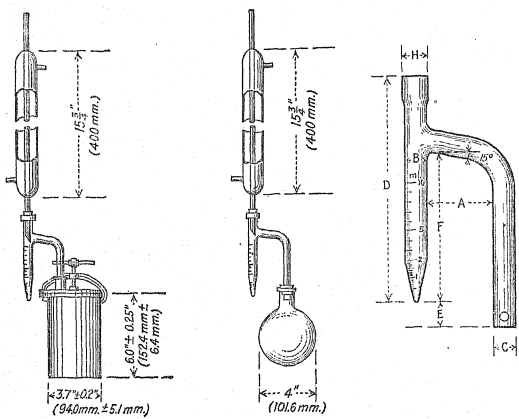
[1] Under the standardization procedure of the Society, this method is under the joint jurisdiction of A.S.T.M. Committee D-2 on Petroleum Products and Lubricants and Committee D-4 on Road and Paving Materials.

[2] Prior to adoption as standard, this method was published as tentative from 1921 to 1924, being revised in 1923 and 1924.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

than 400 mm. (15¾ in.) in length, with an inner tube 9.5 to 12.7 mm. (⅜ to ½ in.) in outside diameter. The end of the condenser to be inserted in the trap shall be ground off at an angle of 30 ±5 deg. from the vertical axis of the condenser.

(*f*) *Trap.*—The trap shall be made of well-annealed glass constructed in accordance with Fig. 1 (*c*) and shall be graduated from 0 to 10 ml. in 0.1-ml. divisions. The error of any indicated capacity shall not be greater than 0.05 ml. The outside diameters should be preferably 2.5 to 3.5 mm. (3/32 to ⅛ in.) greater than the inside diameters specified.



A	45 to	55 mm	1¾ to	2⅛ in.
B (Inside Diameter)	14 to	16 mm	9/16 to	5/8 in.
C (Inside Diameter)	12 to	16 mm.	½ to	5/8 in.
D	150 to	170 mm.	6 to	6¾ in.
E	25 to	38 mm.	1 to	1½ in.
F	100 to	108 mm.	4 to	4¼ in.
H (Inside Diameter)	18 to	19 mm.	11/16 to	¾ in.

FIG. 1.—Apparatus for Determining Water.

Solvent[3a]

3. (*a*) The solvent used when testing petroleum products or bituminous materials derived from petroleum shall be petroleum naphtha and shall conform to the following distillation requirements:

5 per cent shall distill at a temperature not below 194 F. (90 C.) nor above 212 F. (100 C.)

90 per cent shall distill below 410 F. (210 C.)

(*b*) The solvent used when testing bituminous materials derived from coal tar, water-gas tar, etc., shall be coal-tar

[3a] Editorially revised in 1941.

naphtha or a light oil and shall conform to the following distillation requirement:

98 per cent shall distill between 248 F. (120 C.) and 482 F. (250 C.)	

(*c*) The distillation requirements specified for the solvents in Paragraphs (*a*) and (*b*) shall be determined in accordance with the Standard Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (A.S.T.M. Designation: D 86) of the American Society for Testing Materials.[3]

Sample

4. The sample shall be thoroughly representative of the material to be tested and the portion of the sample used for the test shall be thoroughly representative of the sample itself. Deviation from this requirement shall not be permitted.

NOTE.—The difficulties in obtaining proper representative samples for this determination are unusually great so that the importance of sampling cannot be too strongly emphasized.

Procedure

5. (*a*) When the sample to be tested contains less than 10 per cent of water, exactly 100 ml. of the material to be tested shall be placed into the still or flask and thoroughly mixed with an equal volume of solvent by swirling, proper care being taken to avoid any loss of material. If the material is measured by volume, an accurate 100-ml. graduated cylinder shall be used and the contents transferred to the still by rinsing with one 50-ml. portion of solvent followed by two successive 25-ml. portions of solvent, the cylinder being allowed to drain each time. When the sample to be tested contains more than 10 per cent of water, the volume of material used shall be decreased to that which will yield somewhat less than 10 ml. of water.

NOTE.—In special cases where the water content exceeds 10 per cent and it is not desirable to reduce the size of the sample to that which will yield somewhat less than 10 ml. of water, a distilling tube receiver graduated from 0 to 25 ml. may be used. This tube shall be graduated from 0 to 2 ml. in 0.1-ml. divisions and from 2 to 25 ml. in 0.2-ml. divisions (see Fig. 3 (*c*) of the Standard Methods of Testing Emulsified Asphalts (A.S.T.M. Designation: D 244) of the American Society for Testing Materials).[3]

(*b*) The connections between the still or flask, trap, and condenser shall be made by means of tight-fitting corks as shown in Figs. 1 (*a*) and (*b*). The end of the condenser inserted in the trap shall be adjusted to that position which will allow the end to be submerged to a depth of not more than 1 mm. below the surface of the liquid in the trap after distillation conditions have been established. When the metal still is used, a heavy paper gasket moistened with the solvent shall be inserted between the lid and flange before attaching the clamp. A loose cotton plug shall be inserted in the top of the condenser tube to prevent condensation of atmospheric moisture in the condenser tube.

(*c*) Heat shall then be applied and so regulated that the condensed distillate falls from the end of the condenser at the rate of from 2 to 5 drops per sec. The ring burner used with the metal still should be placed about 3 in. above the bottom of the still at the beginning of the distillation and gradually lowered as the distillation proceeds.

(*d*) The distillation shall be continued at the specified rate until no water is visible on any part of the apparatus except at the bottom of the trap. This operation usually requires less than an

hour. A persistent ring of condensed water in the condenser tube shall be removed by increasing the rate of distillation for a few minutes.

Calculations

6. The volume of condensed water measured in the trap at room temperature multiplied by 100 and divided by the volume of the sample used shall be the percentage of water and shall be reported as "- - - - per cent water by volume, A.S.T.M. Method D 95."

Reproducibility of Results

7. Duplicate determinations of water by this method should not differ from each other by more than one division on the trap.

Recommended Practice for

BITUMINOUS PAVING PLANT INSPECTION[1]

A.S.T.M. Designation: D 290 – 39

ADOPTED, 1939.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 290; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This recommended practice defines the authority and duties of the inspector at a bituminous paving plant.

Definitions

2. (*a*) *Engineer.*—Throughout this recommended practice the term engineer shall be construed to mean the engineer in responsible charge of the work or his duly recognized or authorized laboratory; or, in the absence of an engineer, the purchaser or his duly authorized laboratory.

(*b*) *Contractor.*—The term contractor shall be construed to mean the party to the contract who has agreed to supply materials and perform work in accordance with the specifications, or his duly authorized representative at the plant.

(*c*) *Laboratory.*—The term laboratory shall be construed to mean any supervising laboratory duly authorized by the engineer to direct and advise the inspector in the discharge of his duties.

(*d*) *Plant Laboratory.*—The term plant laboratory shall be construed to mean the laboratory operated by or under the direction of the plant inspector at the plant.

RESPONSIBILITIES AND GENERAL DUTIES OF INSPECTOR

Authority

3. The inspector shall be provided with written authority from the engineer to enable him to insist on the fulfillment of specifications covering materials, plant procedure, and products; and the rejection of such materials, procedure, and products failing to conform to the specifications. The inspector shall have available a copy of the specifications applying to the particular project, and shall be furnished immediately in writing with copies of all modifications, amendments, and instructions affecting the product of the plant. The inspector shall recognize the right of the contractor to use such apparatus, methods, and personnel as he deems proper, provided

[1] Under the standardization procedure of the Society, this recommended practice is under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] Prior to adoption as standard, this recommended practice was published as tentative from 1928 to 1939, being revised in 1939.

TABLE I.—INDEX OF SAMPLING BITUMINOUS PAVING MATERIALS.

Material	Method	Quantity	When Collected	By Whom Tested
Asphalt Sand	Tentative Methods of Sampling Stone, Slag, Gravel, Sand, and Stone Block for Use as Highway Materials (A.S.T.M. Designation: D 75) of the American Society for Testing Materials[a]	5 lb.	Each car or boat and truck deliveries by special instructions from preliminary or first shipments from given source, or when source or supply changes, or if change occurs in materials received	Laboratory
			Daily from subsequent shipments, and sand in use from stock pile	Plant Laboratory
Screenings	Same as for asphalt sand	5 lb.	Same as for asphalt sand	Same as for asphalt sand
Crushed Stone	Same as for asphalt sand	15 to 20 lb.	Same as for asphalt sand	Same as for asphalt sand
Filler	Composite from eight to ten sacks, mixing and quartering	5 lb.	Same as for asphalt sand	Same as for asphalt sand
Heated Aggregates	By use of shovel as aggregate flows from storage bin	5 to 20 lb., depending on whether coarse or fine aggregate	At least daily	Plant Laboratory
Asphalt Cement A	Tentative Methods of Sampling Bituminous Materials (A.S.T.M. Designation: D 140) of the American Society for Testing Materials[a]	1 qt.	Each car or boat	Laboratory
Asphalt Cement B	In metal container from valve over asphalt bucket on plant. Mixed and poured into 3-oz. can	3 oz.	Daily[a]	Plant Laboratory and Laboratory
Crude Asphalt	Tentative Methods of Sampling Bituminous Materials (A.S.T.M. Designation: D 140) of the American Society for Testing Materials[a]	1 qt.	Each car or boat	Laboratory
Refined Asphalt		1 qt.	Each car or boat	Laboratory
Flux		1 qt.	Each car or boat	Laboratory
Finished Mixtures A. To determine average daily analysis of mixture	Composite from number of batches	4 oz., min., for sheet asphalt; 5 lb., min., for asphaltic concrete or binder	Daily	Laboratory
Finished Mixtures B. For determination of uniformity of individual or of different batches	From individual batches	4 oz., min., for sheet asphalt; 5 lb., min., for asphaltic concrete or binder	As directed	Laboratory
Asphalt Block	Select blocks representative of production	2 blocks	Daily	Laboratory

[a] When fluxing at paving plants, preliminary 3-oz. samples shall also be taken from each melting kettle, when ready and completely mixed. This is in addition to daily 3-oz. sample as provided above.

[a] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

it is within the specifications. Any appeal on the part of the contractor from rejections shall be made in writing to the engineer, with a copy to the inspector, unless otherwise provided in the contract.

Cooperation

4. It shall be the duty of the plant inspector to cooperate in every reasonable way with the contractor so as to assist in obtaining economical and efficient plant operation; and with the pavement inspector in all plant matters relating to the economical production of a satisfactory material conforming to the specifications.

Duties

5. In addition to the responsibilities specified in Sections 3 and 4, the duties of the plant inspector shall be:

(*a*) To ascertain that the plant is equipped and operated at all times in conformity with the specifications (see Section 6),

(*b*) To collect and submit to the laboratory or to test, as directed, samples to be used at the plant (see Sections 7 to 13, inclusive, and Table I),

(*c*) To control the proportions and temperatures of mixtures and of materials within such limits as are assigned to him, and to insure that the products of the plant are otherwise in accordance with the specifications (see Sections 14 to 18),

(*d*) To conduct required plant-laboratory tests of materials (see Sections 19 to 24),

(*e*) To record the output of the plant, and, as required, the delivery and consumption of materials at the plant (see Section 25),

(*f*) To fulfill other assigned duties, (see Section 26), and

(*g*) To submit daily reports (see Sections 27 and 28).

PLANT EQUIPMENT AND OPERATION

Plant Equipment and Operation

6. The inspector shall ascertain as early as possible that the plant is equipped in accordance with specifications and capable of producing a mixture conforming to the requirements of the specifications. He shall also determine if the liners, gate, and blades of the mixer are in satisfactory condition for efficient mixing. He shall make all necessary determinations of the accuracy of scales, using standard weights, and of the volume of various containers if used in the proportioning of mixtures. During the operation of the plant, he shall check at frequent intervals the accuracy of various scales used in determining the weight of each material. He shall examine sieves used in separating heated aggregates for bin storage to see that the sieve openings are of the proper size, and that the various sieve sections are properly placed over the bins.

SAMPLING

Sampling

7. As far as possible in advance, and preferably at least three to five days before beginning work on each contract, samples of each material to be used in the mixtures shall be collected by the inspector and submitted to the laboratory or tested by him as directed as shown in Table I. Should the sources of supply of any material received change from that represented by the first sample taken by the inspector, new samples shall be submitted by the inspector.

Marking

8. Beginning at the start of operations on a given contract, the inspector shall assign class designations to each class of material being sampled, and each sample

selected shall be numbered serially in that class, continuing numerically until the completion of the work. Care shall be taken that the series of numbers is not broken or numbers duplicated. All samples sent to the laboratory shall be marked in such a manner as to insure complete identification. Letters and numbers may be scratched or punched clearly on tin cans, and marked with ink or paint on other sample containers or on sections of pavement.

Asphalt Cement

9. (*a*) Daily control samples of asphalt cement and mixtures shall be selected and, at the end of the day's work, forwarded to the laboratory without delay.

(*b*) Daily samples of asphalt cement which the inspector has tested for penetration shall be forwarded to the laboratory.

Crude Asphalt, Refined Asphalt, and Flux

10. When forwarding samples of crude asphalt, refined asphalt, and flux to the laboratory, in the inspection covering the manufacture of asphalt blocks, information shall also be furnished relative to the proportions by weight being used so that proper combination can be made for analysis and check determinations.

Sheet Asphalt Surface Mixture

11. Daily samples of sheet asphalt surface mixtures shall be obtained by the inspector for the laboratory. Each sample shall consist of small quantities from not less than ten, and preferably more, separate batches. The portions selected shall be molded into a container provided for that purpose. Care shall be taken in selecting and after collection of these samples to prevent contamination by dust or other foreign material. It is desirable to scrape away the top material in a batch before sampling to prevent any contamination.

Bituminous Concrete

12. (*a*) *Control Samples.*—Daily samples of bituminous concrete mixtures shall be taken by the inspector, using either method (1) or (2) as follows:

(1) Take small portions from a number of batches and store them on a metal plate until near the end of the day. At the end of this time heat the entire mass, taking care not to apply too high a temperature, mix thoroughly, and select the sample in small portions from this mass.

(2) Collect portions from one or more batches on a large shovel, mix thoroughly while still warm, and place a representative scoopful directly in a container. This operation shall be repeated as often as possible to represent a maximum number of batches.

Care shall be taken to avoid segregation of either stone or bituminous mortar, and in selecting the portions of the sample care shall be taken to scrape away the top material in a load so as to avoid dust or any segregated unrepresentative mixture. Precautions shall also be taken to prevent contamination, by dust or other foreign material, of the sample after collection.

(*b*) *Uniformity Samples.*—Samples shall be taken by the inspector, as directed, to show the uniformity of the mixture by either selecting three separate portions from separate distinct spots in the batch, or by selecting a representative sample from different individual batches throughout a day's run. The samples collected shall be placed in separate containers and each container shall be properly identified and distinctly marked "Uniformity Sample" furnishing also the temperature and length of time the batch was mixed.

Rejected Materials

13. Samples of all materials rejected by the inspector shall be forwarded to the laboratory.

PREPARATION OF MIXTURES

Proportioning Materials

14. Within the limits of the specifications, the inspector shall receive instructions from the engineer in regard to the desired proportion of each ingredient that enters into a mixture and the range of proportions within which he should work. It is advisable, whenever possible, to have entire loads of the mixture weighed, as a further check on the weight or volumetric proportioning being used in the plant.

Volumetric Proportioning

15. (*a*) *Dry Aggregates.*—When dry, heated sand and stone are measured by volume, their weights per cubic foot, hot, shall be determined in order to calculate the proportions. The weight per cubic foot of the filler shall also be determined, at the approximate temperature of use, for calculating the correct amount to be added.

(*b*) *Wet Aggregates.*—When wet sand, stone, and screenings are being measured by volume, the moisture content of each shall be determined by drying a representative sample and calculating the percentage of moisture. Deductions for moisture shall be made from the weights of each material forming the total batch in calculating the required amount of asphalt cement. The percentage of moisture in aggregates shall always be entered on the daily report. The determination shall be made frequently on sand and fine screenings. Loss of fine material in the drying process shall be compensated for by the use of additional filler in proportioning.

Proportioning by Weight

16. (*a*) Asphalt cement shall always be measured by weight. The actual percentage of bitumen in the asphalt cement should be ascertained from the laboratory in order to determine the proportion of asphalt cement to be used in the mixture.

(*b*) The inspector should note whether the scales on the mixing platform are being handled carefully, taking care that the aggregate hoppers and asphalt bucket swing freely during the weighing and that the beam is brought to a floating position. He should note whether the asphalt bucket is drained completely for each batch, and check the tare weight of the empty asphalt bucket at frequent intervals so as to compensate for the amount of asphalt clinging to the bucket after each pouring. Particular attention shall be paid to this point in cool weather and at times when there has been a long interval between loads.

Combining Aggregates

17. (*a*) The inspector shall give particular attention to the combination of aggregates before they enter the drier, and especially to the combination of fine aggregates. Where the proportions of the final mixture are dependent upon the supply of aggregates to a belt conveyor or elevator, he shall see that the supply of each aggregate to the conveyor is continuous and uniform. When two or more kinds of aggregate are combined on the ground and measured in such containers as wheelbarrows, which must be of equal volumes, the containers shall be struck off to a level surface. Irregular heaping shall not be permitted.

(*b*) The inspector shall make certain that the mixer operator allows mineral aggregates to become thoroughly mixed before the addition of asphalt cement, and that the time of mixing is sufficient to prepare a uniform mixture.

Temperature of Materials

18. (*a*) The inspector shall check the temperature of all ingredients of the mixture, especially mineral aggregates, and of the mixtures, as frequently as possible. He should assist plant employees in every way in keeping temperatures uniform by notifying the plant foreman of any marked changes that he may observe. Should the aggregates be of improper temperature, mixing operations shall be discontinued until aggregates of the proper temperature are obtained.

(*b*) The desired temperatures at the plant will depend upon the kind of mixture being prepared, the type of asphalt being used, weather conditions, and the length of haul. The inspector should receive instructions as to the temperature range desired for each contract.

PLANT TESTING OF MATERIALS

Sand

19. The sands shall be tested daily in accordance with the Standard Method of Test for Sieve Analysis of Fine and Coarse Aggregates (A.S.T.M. Designation: C 136) of the American Society for Testing Materials.[3] When two or more grades of sand are being received for use in combination, separate mechanical analysis shall be made on shipments of each variety and the resultant combination calculated and shown on the daily report. The proportions in which sands are combined shall also be shown on the report.

Filler

20. Samples of the thoroughly dry filler shall be tested in accordance with the Standard Method of Test for Sieve Analysis of Mineral Filler (A.S.T.M. Designation: D 546) of the American Society for Testing Materials.[3]

Screenings

21. The mechanical analysis of screenings shall be determined on a dried sample of not less than 250 g., and if the screenings are very coarse or contain a considerable percentage of particles larger than $\frac{1}{2}$ in. in diameter, an amount up to 500 g. may be used. The sample shall be sieved on a No. 10 (2000-micron) sieve and the percentages retained on and passing this sieve shall be determined. If the amount passing the No. 10 sieve is much greater than 100 g., a portion of it, after thorough mixing, may be selected for sieve analysis by quartering and then tested as previously specified in Section 19. The percentages retained on each sieve shall be calculated on the basis of the original sample. The portion retained on the No. 10 sieve shall be sieved over various coarser sieves as may be required.

Stone

22. Coarse aggregate shall be tested for grading in accordance with the Standard Method of Test for Sieve Analysis of Fine and Coarse Aggregates (A.S.T.M. Designation: C 136) of the American Society for Testing Materials.[3] When stone is being used from a large stock pile, frequent mechanical analyses shall be made as the work progresses.

Asphalt Cement

23. (*a*) The inspector shall make daily penetration tests of each tank of asphalt cement used in the day's work. The sample shall be placed in 3-oz. tin boxes and the test made in accordance with the Standard Method of Test for Penetration of Bituminous Materials (A.S.T.M. Designation: D 5) of the American Society for Testing Materials.[3] After the test, all water shall be drained from the samples and the boxes closed with

ASPHALT PLANT REPORT

State or County | City, Twp. or Boro

Street, Route, Appl. | Sec. No. or Block

Date.....19....Weather.....Temp. A.M.....P.M.....F. A. P. No .....Eng. Dist.....
Type of Const.....Type of Plant.....Location of Plant.....
Contractor.....Amount of Contract.....sq. yd.
Binder or S. C. Sta.....to Sta.....Lineal ft. S. C. or Binder.....
W. S. or B. C. Sta.....to Sta.....Lineal ft. W. S. or B. C.....

Course	Temps. Max.	Temps. Min.	Temps. Ave.	No. Batches	Hr. in Operation	Sq. Yd. Laid
Binder or S. C.						
W. S. or B. C.						

Materials	Kind or Brand	Tons on Hand	Tons Used
Refined Asphalt			
Flux			
Asphaltic Cement			
Filler (Dust)			
Portland Cement			
Sand			
Gravel			
Crushed Stone			
Screenings			
Coal			
Coke			
Fuel Oil (gal.)			
Lub. Oil (gal.)			

Binder or S. C., lb. per sq. yd.....
W. S. or B. C., lb. per sq. yd.....
Total, lb. per sq. yd.....
No. of sq. yd. laid to date, Completed Pav't.....
No. of sq. yd. laid today, Completed Pav't.....
No. of sq. yd. laid, total, Completed Pav't.....

Plant Labor	No.	Hours	Total Hours
Foremen			
Engineers			
Skilled Labor			
Unskilled Labor			
Trucks			
Total Hours			

Sampled From	Sand				Stone				Screenings		Penetrations No.	Penetrations Test	Samples to Laboratory	
Per Cent Passing: No. 200													W. S.	
No. 80													B. C.	
No. 40													S. C.	
No. 10													A. C.	
No. 4													R. A.	
½ in.														
¾ in.													Filler	
1 in.													Sand	
1½ in.													Screenings	
Ret. on 1½ in.													Stone	
Total														

Temperatures		Tank No. 1 A. C.	Tank No. 2 A. C.
Max.			
Min.			
Avg.			

BATCH WEIGHTS AND PERCENTAGE OF MIXTURES

Material	Binder (lb.)	Per Cent	W. S. (lb.)	Per Cent	B. C. (lb.)	Per Cent	S. C. (lb.)	Per Cent	
A. C.									
Filler (Dust)									
Sand									
Screenings									
Stone									
Total									

Time arrived.....
Beginning of operation.....
Time taken for lunch.....
Time departed.....

Record of Official Inspections.....
Calculations.....Notes to Lab.....Remarks.....
Shown on reverse side (check items to which reference is made)

.....Inspector.

NOTE:
A. C. = Asphalt Cement.
B. C. = Bituminous Concrete.
R. A. = Refined Asphalt.
S. C. = Seal Coat.
W. S. = Wearing Surface.

tightly fitting lids for shipment to the laboratory.

(*b*) A working range of ten points penetration for the asphalt cement will usually be set by the specifications for each contract, and if results should show a deviation beyond this range a check determination shall be made. The inspector should be instructed as to what action he should take when the penetration of asphalt cement is not within the prescribed limits.

Heated Aggregates

24. Samples of aggregates which have passed through the drying and heating process shall be taken and tested at least once each day, and more frequently if necessary, to determine the uniformity of bin materials. Samples may be secured by passing a shovel or pan quickly through the stream of hot material as it flows from the storage bin. All tests of these materials shall be recorded on the plant report. The results will show: (1) when blended materials are being used, whether mixing is being carried on satisfactorily, and (2) whether separation of aggregates by sizes has been complete. If the dried sand is contaminated with stone, steps shall be taken to determine the cause and to make correction.

REPORT

Production of Plant

25. The inspector shall record daily the time and hours of operation of the plant, the number of batches and number of loads of each type of mixture produced, and also the total amounts of each material used during the day and on hand at the end of the day. At times, the inspector may be required to record the locations at which mixtures were laid on the road.

Miscellaneous Data

26. When obtainable, the inspector may be required to report cost data, inincluding the hours of work for each classification of labor, trucks, superintendents, etc., and the rates of compensation.

Daily Reports

27. At the close of each working day, whether the plant is in operation or not, the inspector shall forward to the engineer and laboratory a consecutively numbered daily report, using a form which gives all the required information. All data pertinent to the work not covered by the form should be given on the reverse side of the report sheet, and communications of exceptional importance only should be sent separately from the report. When work is suspended for several days, the inspector may not be required to submit reports daily, but the last report should state why and for what periods work has been discontinued. The inspector shall retain copies of these reports and communications and, in addition, keep a daily record of all matters pertaining to his work, particularly as to his action in acceptance or rejection of materials with reasons therefor, instructions or suggestions to the contractor, and receipt of instructions from the engineer or laboratory.

Report Form

28. A standard form of report is not practicable for all conditions and types of pavement, but the report form appearing at the end of this recommended practice is a typical form showing the kind of information which should be included; additional data may be called for as required.

Standard Definitions of

TERMS RELATING TO MATERIALS FOR ROADS AND PAVEMENTS[1]

A.S.T.M. Designation: D 8 – 33

ADOPTED, 1912; REVISED, 1915, 1918, 1932, 1933.

This Standard of the American Society for Testing Materials is issued under the fixed designation D 8; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

BITUMINOUS MATERIALS

Relating in General to Bituminous Materials

Bitumens.—Mixtures of hydrocarbons of natural or pyrogenous origin, or combinations of both, frequently accompanied by their non-metallic derivatives, which may be gaseous, liquid, semisolid, or solid, and which are completely soluble in carbon disulfide.

Bituminous.—Containing bitumen or constituting the source of bitumen.

Bituminous Emulsion.—A liquid mixture in which minute globules of bitumen are held in suspension in water or a watery solution.

Cut-Back Products.—Petroleum or tar residuums which have been fluxed with distillates.

Fluxes.—Bituminous materials, generally liquid, in which the predominating constituent is bitumen, used in combination with asphalts for the purpose of softening the latter.

Liquid Bituminous Materials.—Those, having a penetration at 25 C. (77 F.), under a load of 50 g. applied for 1 sec., of more than 350.

Semisolid Bituminous Materials.—Those having a penetration at 25 C. (77 F.), under a load of 100 g. applied for 5 sec., of more than 10, and a penetration at 25 C. (77 F.), under a load of 50 g. applied for 1 sec., of not more than 350.

Solid Bituminous Materials.—Those having a penetration at 25 C. (77 F.), under a load of 100 g. applied for 5 sec., of not more than 10.

Relating Specifically to Petroleum or Asphalts

Asphalts.—Black to dark-brown solid or semisolid cementitious materials which gradually liquefy when heated, in which the predominating constituents are bitumens all of which occur in the solid or semisolid form in nature or are obtained by refining petroleum, or which are combinations of the bitumens mentioned with each other or with petroleum or derivatives thereof.

[1] Under the standardization procedure of the Society, these definitions are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

Artificial Asphalt.—Recommended that the use of term be discontinued.

Asphalt Cement.—A fluxed or unfluxed asphalt specially prepared as to quality and consistency for direct use in the manufacture of bituminous pavements, and having a penetration at 25 C. (77 F.) of between 5 and 250, under a load of 100 g. applied for 5 sec.

Liquid Asphalt.—This is a trade term not subject to definition.

Native Asphalt.—Asphalt occurring as such in nature.

Road Asphalt.—This is a trade term not subject to definition.

Rock Asphalt.—Sandstone or limestone naturally impregnated with asphalt.

Petroleum.—Liquid bitumen occurring as such in nature.

Blown Petroleums.—Semisolid or solid products produced primarily by the action of air upon liquid native bitumens which are heated during the blowing process.

Topped Petroleum.—Petroleum deprived of its more volatile constituents.

Asphaltenes.—The components of the bitumen in petroleums, petroleum products, malthas, asphalt cements, and solid native bitumens, which are soluble in carbon disulfide but insoluble in paraffin naphthas.

Carbenes.—The components of the bitumen in petroleums, petroleum products, malthas, asphalt cements, and solid native bitumens, which are soluble in carbon disulfide but insoluble in carbon tetrachloride.

Relating Specifically to Tars and Pitches

Tars.—Black to dark-brown bituminous condensates which yield substantial quantities of pitch when partially evaporated or fractionally distilled, and which are produced by destructive distillation of organic material, such as coal, oil, lignite, peat, and wood.

Coal Tar.—Tar produced by the destructive distillation of bituminous coal.

Gas-House Coal Tar.—Coal tar produced in gas-house retorts in the manufacture of illuminating gas from bituminous coal.

Coke-Oven Tar.—Coal tar produced in by-product coke ovens in the manufacture of coke from bituminous coal.

Oil-gas Tars.—Tars produced by cracking oil vapors at high temperatures in the manufacture of oil gas.

Water-Gas Tars.—Tars produced by cracking oil vapors at high temperatures in the manufacture of carburetted water-gas.

Dehydrated Tars.—Tars from which all water has been removed.

Refined Tar.—Tar freed from water by evaporation or distillation which is continued until the residue is of desired consistency; or a product produced by fluxing tar residuum with tar distillate.

Dead Oils.—Oils with a density greater than water which are distilled from tars.

Pitches.—Black or dark-brown solid cementitious residues which gradually liquefy when heated and which are produced by the partial evaporation or fractional distillation of tars.

Straight-run Pitch.—A pitch run to the consistency desired, in the initial process of distillation, without subsequent fluxing.

Free Carbon in Tars.—Organic matter which is insoluble in carbon disulfide.

Relating Specifically to Tests

Normal Temperature.—As applied to laboratory observations of the physical characteristics of bituminous materials, is 25 C. (77 F).

Consistency.—The degree of solidity or fluidity of bituminous materials.

Fixed Carbon.—The organic matter of the residual coke obtained upon burning hydrocarbon products in a covered vessel in the absence of free oxygen.

Penetration.—The consistency of a bituminous material expressed as the distance that a standard needle vertically penetrates a sample of the material under known conditions of loading, time and temperature. Where the conditions of test are not specifically mentioned, the load, time, and temperature are understood to be 100 g., 5 sec., and 25 C. (77 F.), respectively, and the units of penetration to indicate hundredths of a centimeter.

Viscosity.—The measure of the resistance to flow of a bituminous material, usually stated as the time of flow of a given amount of the material through a given orifice.

NONBITUMINOUS MATERIALS

Relating in General to Nonbituminous Materials

Aggregate.—The inert material, such as sand, gravel, shell, slag, or broken stone or combinations thereof, with which the cementing material is mixed to form a mortar or concrete.

Matrix.—The binding material or mixture of binding material and fine aggregate in which the large aggregate is embedded or held in place.

Crusher-Run.—The total unscreened product of a stone crusher.

Stone Chips.—Small angular fragments of stone containing no dust.

Tailings.—Stones which after going through the crusher do not pass through the largest openings of the screen.

Rubble.—Rough stones of irregular shapes and sizes, broken from larger masses either naturally or artificially, as by geological action, in quarrying, or in stone cutting or blasting.

Relating Specifically to Materials

Bank Gravel.—Gravel found in natural deposits, usually more or less intermixed with fine material, such as sand or clay, or combinations thereof; gravelly clay, gravelly sand, clayey gravel and sandy gravel, indicate the varying proportions of the materials in the mixture.

Clinker.—Generally a fused or partly fused by-product of the combustion of coal, but also including lava and portland-cement clinker, and partly vitrified slag and brick.

Chert.—Compact siliceous rock formed of calcedonic or opaline silica, or both.

Granite.—A granitoid igneous rock consisting of quartz, orthoclase, more or less oligoclase, biotite and muscovite.

Granitoid.—A textural term to describe those igneous rocks which are entirely composed of recognizable minerals.

Slag.—Fused or partly fused compounds of silica in combination with lime or other bases, resulting in secondary products from the reduction of metallic ores.

Soil.—A mixture of fine earthy material with more or less organic matter resulting from the growth and decomposition of vegetation or animal matter.

Relating Specifically to Tests

Mesh.—The square opening of a sieve.

Screen.—In laboratory work an apparatus, in which the apertures are circular, for separating sizes of material.

Sieve.—In laboratory work an apparatus, in which the apertures are square, for separating sizes of material.

Standard Method of

PREPARING SOIL SAMPLES FOR MECHANICAL ANALYSIS AND DETERMINATION OF SUBGRADE SOIL CONSTANTS[1]

A.S.T.M. Designation: D 421 - 39

ADOPTED, 1939.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 421; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method covers the preparation of soil samples as received from the field for mechanical analysis and the determination of the subgrade soil constants.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Balance.*—A balance sensitive to 0.1 g.

(*b*) *Mortar.*—A mortar and rubber-covered pestle suitable for breaking up the aggregations of soil particles.

(*c*) *Sieves.*—A series of sieves, of square mesh woven wire cloth, conforming to the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials.[3] The sieves required are as follows:

No. 4 (4760-micron)
No. 10 (2000-micron)
No. 40 (420-micron)
No. 200 (74-micron)

(*d*) *Sampler.*—A riffle sampler or sample splitter, for quartering the samples.

Sampling

3. The soil sample as received from the field shall be dried thoroughly in the air and the aggregations shall then be thoroughly broken up in the mortar with a rubber-covered pestle. A representative test sample of the amount required to perform the desired tests shall then be selected by the method of quartering or by the use of a sampler. The amounts of material required to perform the individual tests are as follows:

(*a*) *Mechanical Analysis.*—For the mechanical analysis, material passing a No. 10 (2000-micron) sieve is required in amounts equal to 115 g. of sandy soils and 65 g. of either silt or clay soils.

(*b*) *Physical Tests.*—For the physical tests, material passing the No. 40 (420-

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-18 on Soils for Engineering Purposes.

[2] Prior to adoption as standard, this method was published as tentative from 1935 to 1939, being revised in 1938.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

micron) sieve is required in total amount equal to 200 g., allocated as follows:

Test	Grams
Liquid limit	30
Plastic limit	15
Centrifuge moisture equivalent	10
Field moisture equivalent	50
Volumetric shrinkage	30
Check tests	65

Preparation of Test Sample

4. (*a*) That portion of the air-dried sample selected for purpose of tests shall be weighed and the weight recorded as the weight of the total test sample uncorrected for hygroscopic moisture. The test sample shall be separated by sieving with a No. 10 (2000-micron) sieve. That fraction retained on the No. 10 sieve shall be ground in a mortar with a rubber-covered pestle until the aggregations of soil particles are broken up into the separate grains. The ground soil shall then be separated into two fractions by sieving with a No. 10 sieve.

(*b*) That fraction retained after the second sieving shall be washed free of all fine material, dried, and weighed. This weight shall be recorded as the weight of coarse material. The coarse material after being washed and dried shall be sieved on the No. 4 (4760-micron) sieve and the weight retained on the No. 4 sieve recorded.

Test Sample for Mechanical Analysis

5. The fractions passing the No. 10 (2000-micron) sieve in both sieving operations shall be thoroughly mixed together, and by the method of quartering or the use of a sampler a portion weighing approximately 115 g. for sandy soils and approximately 65 g. for silt and clay soils shall be selected for mechanical analysis.

Test Sample for Subgrade Soil Constants

6. The remaining portion of the material passing the No. 10 (2000-micron) sieve shall then be separated into two parts by means of a No. 40 (420-micron) sieve. The fraction retained on the No. 40 sieve shall be discarded. The fraction passing the No. 40 sieve shall be used for the determination of the subgrade soil constants.

Standard Method of

MECHANICAL ANALYSIS OF SOILS[1]

A.S.T.M. Designation: D 422 – 39

ADOPTED, 1939.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 422; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method describes a procedure for the quantitative determination of the distribution of particle sizes in soils.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Balance.*—A balance sensitive to 0.1 g.

(*b*) *Stirring Apparatus.*—A special stirring apparatus (Fig. 1) with replaceable stirring paddle (Fig. 2) and specially designed dispersion cup of either of the designs shown in Fig 3.

(*c*) *Hydrometer.*—A hydrometer graduated in grams of soil per liter of suspension (Fig. 4, *A*) *or* a hydrometer with special shape bulb, graduated in specific gravity, having a range of 0.995 to 1.050 sp. gr. and reading 1.000 at 67 F. (19.4 C.) (Fig. 4, *B*).

(*d*) *Glass Graduate.*—A glass graduate 18 in. in height and 2½ in. in diameter, and graduated for a volume of 1000 ml.

(*e*) *Thermometer.*—A Fahrenheit thermometer accurate to 1 F. (0.5 C.).

(*f*) *Sieves.*—A series of sieves, of square mesh woven wire cloth, conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials.[3] The sieves required are as follows:

No. 20 (840-micron)
No. 40 (420-micron)
No. 60 (250-micron)
No. 140 (105-micron)
No. 200 (74-micron)

(*g*) *Water Bath.*—A water bath for maintaining the soil suspension at a constant temperature during the hydrometer analysis. A satisfactory device is an insulated zinc tank which maintains the temperature of the suspension of faucet-water temperature. Such a device is illustrated in Fig. 5.

(*h*) *Beaker.*—A tall-form beaker of 400-ml. capacity.

Sample

3. A sample of soil shall be selected by the method of quartering or by the

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-18 on Soils for Engineering Purposes.

[2] Prior to adoption as standard, this method was published as tentative from 1935 to 1939, being revised in 1938.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

A "tentative revision" of this standard appears in the Tentative Revisions of Standards section at the back of this book.

use of a sampler from the material passing the No. 10 (2000-micron) sieve. The weight of each sample shall be approximately 115 g. for sandy soils and 65 g. for silt or clay soils and shall be obtained in accordance with the Standard Method of Preparing Soil Samples for Mechanical Analysis and Determination of Subgrade Soil Constants (A.S.T.M. Designation: D 421) of the American Society for Testing Materials.[3]

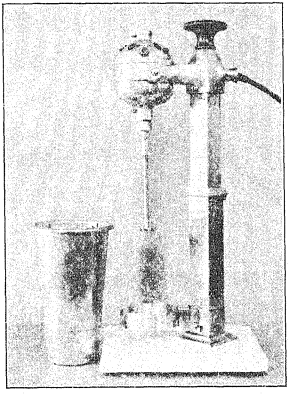


FIG. 1.—Stirring Apparatus.

HYGROSCOPIC MOISTURE

Hygroscopic Moisture

4. A 10-g. portion of the sample selected for mechanical analysis shall be used for the determination of the hygroscopic moisture. This portion of the sample shall be weighed, dried to constant weight in an oven at 110 C., weighed, and the results recorded.

HYDROMETER TEST

Dispersion of Soil Sample

5. The remainder of the sample selected for mechanical analysis shall be weighed and dispersed by one of the two methods *A* or *B* described below. The method to be used depends on the plasticity index of the soil determined in accordance with the Standard Method of Test for Plastic Limit and Plasticity Index of Soils (A.S.T.M. Designation: D 424) of the American Society for Testing Materials.[3]

Method A: For Soils Having a Plasticity Index of 20 or Less.—The soil shall

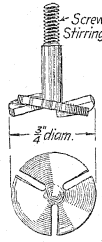


FIG. 2.—Detail of Stirring Paddle.

be placed in a tall-form beaker, and 200 ml. or more of distilled water added slowly and while stirring constantly until the soil is thoroughly wetted. The mixture shall be allowed to stand for a period of at least 18 hr. It shall then be washed into the special dispersion cup and distilled water added until the cup is within 2 in. of being filled. A deflocculating agent, 20 ml. of a solution of sodium silicate crystals ($Na_2SiO_3 \cdot 9H_2O$), shall be added. The density of this solution shall be that indicated by one of the following hydrometer readings: hydrometer *A*, 36.5 at 67 F. (19.4 C.);

hydrometer *B*, 1.023 at 67 F. (19.4 C.); Baumé hydrometer, 3 deg. at 76 F. (24.4 C.). The contents of the cup shall be mixed by the special stirring apparatus (Note) for a period of 1 min.

Method B: For Soils Having a Plasticity Index Greater Than 20.—The soil shall be placed in a tall-form beaker, and 100 ml. of hydrogen peroxide (6 per cent) added slowly and while stirring constantly until the soil is thoroughly wetted. The beaker shall then be covered with a watch glass and placed in an oven at 110 C. for 1 hr. The purpose of the peroxide is to assist in the dispersion rather than to remove the organic matter. The beaker shall then be removed from the oven, 100 ml. of distilled water added, and the mixture allowed to stand for at least 18 hr. The mixture shall then be washed into the special dispersion cup, and dispersed (Note) in the same manner and for the same length of time as in method *A*.

NOTE.—It is important in all cases to see that the paddle on the dispersion machine is replaced as soon as it shows signs of wear.

Hydrometer Test

6. (*a*) After dispersion, the mixture shall be transferred to the glass graduate, and distilled water having the same temperature as the constant temperature bath added until the mixture attains a volume of 1000 ml. The graduate containing the soil suspension shall then be placed in the constant temperature bath. The suspension shall be stirred frequently with a glass rod to prevent settlement of particles in suspension. When the soil suspension attains the temperature of the bath the graduate shall be removed and its contents thoroughly shaken for 1 min., the palm of the hand being used as a stopper over the mouth of the graduate.

(*b*) At the conclusion of this shaking the time shall be recorded, the graduate

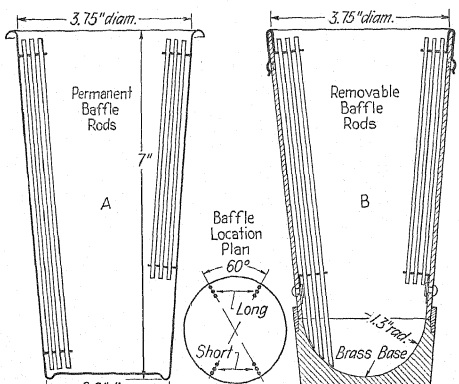


FIG. 3.—Dispersion Cups.

placed in the bath, and readings taken with the hydrometer at the end of both 1 and 2 min. The hydrometer shall be read at the top of the meniscus formed by the suspension around its stem. If hydrometer *A* is used, it shall be read to the nearest 0.5 g. per liter. Hydrometer *B* shall be read to the nearest

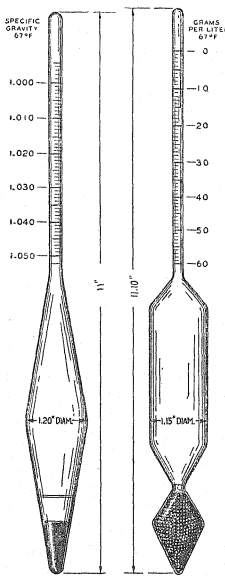


FIG. 4.—Hydrometers.

0.0005 specific gravity. Subsequent readings shall be taken at intervals of 5, 15, 30, 60, 250, and 1440 min. after the beginning of sedimentation. Readings on the thermometer placed in the constant temperature bath shall be made coincidentally with the hydrometer readings and recorded.

(*c*) After each reading except the 1-min. reading, the hydrometer shall be very carefully removed from the soil suspension in such a manner as to cause no disturbance in the suspension, wiped clean, and laid aside. About 15 or 20 sec. before the time for a reading it shall again be slowly and carefully placed in the soil suspension. The reading shall not be taken until the hydrometer has come to rest.

SIEVE ANALYSIS

Sieve Analysis

7. At the conclusion of the final reading the suspension shall be washed on a No. 200 (74-micron) sieve. That fraction retained on the No. 200 sieve shall be dried and then analyzed in a nest of sieves consisting of one each of the Nos. 20, 40, 60, 140, and 200 which conform to Section 2(*f*).

CALCULATIONS

Hygroscopic Moisture

8. The hygroscopic moisture shall be expressed as a percentage of the weight of the oven-dried soil and shall be determined as follows:

$$\text{Hygroscopic moisture} = \frac{W - W_1}{W_1} \times 100$$

where:

W = weight of air-dried soil, and
W_1 = weight of oven-dried soil.

To correct the weight of the air-dried sample of hygroscopic moisture the given value shall be multiplied by the expression:

$$\frac{100}{100 + \text{percentage of hygroscopic moisture}}$$

Coarse Material

9. (*a*) The percentage of coarse material shall be obtained from the data recorded during the preparation of the

sample in accordance with the Standard Method of Preparing Soil Samples for Mechanical Analysis and Determination of Subgrade Soil Constants (A.S.T.M. Designation: D 421) of the American Society for Testing Materials.[3]

(*b*) The coarse material shall be determined as follows: From the weight of the air-dried total test sample subtract the weight of the oven-dried fraction retained on the No. 10 (2000-micron) sieve. The difference is assumed to equal the weight of the air-dried fraction passing the No. 10 sieve (Note 1). The weight of the fraction passing the No. 10 sieve shall be corrected for hygroscopic moisture as indicated in Section 8. To this value shall be added the weight of the oven-dried fraction retained on the No. 10 sieve to obtain the weight of the total test sample corrected for hygroscopic moisture. The fractions retained on both the No. 4 (4760-micron) and the No. 10 (2000-micron) sieve shall be expressed as percentages of the corrected weight of the total test sample (Note 2).

NOTE 1.—According to this assumption, no hygroscopic moisture is contained in the air-dried particles retained on the No. 10 sieve, when as a matter of fact a small percentage of moisture may be present in this fraction. This amount of moisture, compared with that held in the pores of the fraction passing the No. 10 sieve, is relatively small. Therefore, any error produced by the assumption as stated may be considered negligible in amount.

NOTE 2: *Example.*—The following illustration is an example of the data used in calculating the hygroscopic moisture and percentage of coarse material:

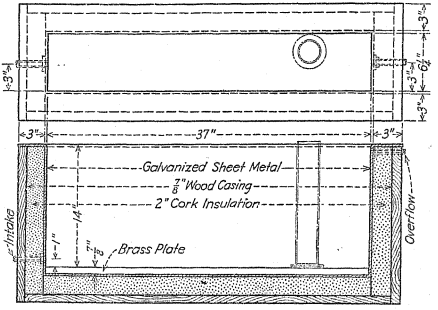


FIG. 5.—Insulated Water Bath.

Hygroscopic Moisture

Weight of air-dried soil, g	15.00
Weight of oven-dried soil, g	14.63
Hygroscopic moisture, percentage of weight of oven-dried soil	2.53
Hygroscopic moisture correction factor	0.975

Coarse Material

Weight of total test sample, air-dried, g	318.3
Weight of washed and oven-dried fraction retained on No. 10 (2000-micron) sieve, g	56.2
Weight of fraction passing No. 10 sieve, air-dried, g	262.1
Weight of fraction passing No. 10 sieve corrected for hygroscopic moisture, g	255.5
Weight of total test sample corrected for hygroscopic moisture, g	311.7
Weight of fraction retained on No. 4 (4760-micron) sieve, oven-dried, g	40 6
Fraction retained on No. 4 sieve, percentage of corrected weight of total test sample	13.0
Fraction retained on No. 10 sieve, percentage of corrected weight of total test sample	18.0

Percentage of Soil in Suspension

10. (*a*) For temperatures of the constant temperature bath other than that at which the hydrometers were cali-

brated, the readings of hydrometer *A* shall be corrected by adding temperature-correction factors such as are shown graphically as Δ R in Fig. 6 (*a*). A temperature-correction curve of this type should be determined experimentally for each hydrometer of type *A* used.

(*b*) Readings of hydrometer *B* shall be corrected by adding temperature-correction factors shown as Δ R in Fig. 6 (*b*). The temperature-correction curve for hydrometer *B* may be determined by plotting the differences between the density of water at the various temperatures and that at 67 F. (19.4 C.) against temperatures from 40 to 100 F. (4.4 to 37.8 C.).

(*c*) The percentage of the dispersed soil in suspension represented by different corrected hydrometer readings depends upon both the amount and the specific gravity of the soil dispersed. The percentage of dispersed soil remaining in suspension may be calculated as follows:

For hydrometer *A*:

$$P = \frac{Ra}{W} \times 100$$

For hydrometer *B*:

$$P = \frac{1606\,(R - 1)a}{W} \times 100$$

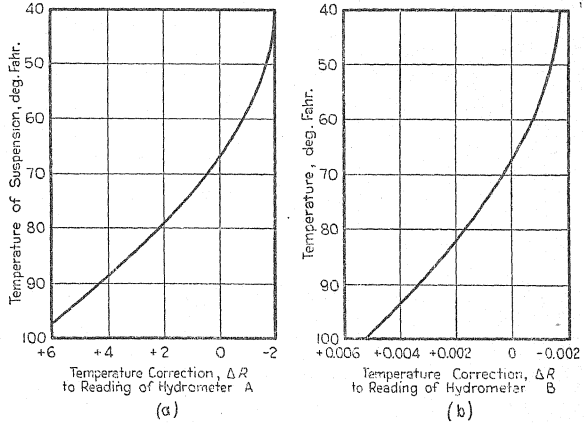


FIG. 6.—Temperature-Correction Curves for Hydrometer Readings.

where:

P = percentage of originally dispersed soil remaining in suspension,

R = corrected hydrometer reading,

W = weight in grams of soil originally dispersed minus the hygroscopic moisture, and

a = constant depending on the density of the suspension.

For hydrometer *A*, the value of *a*, for a specific gravity *G* and a water density at 67 F. (19.4 C.) of 0.9984, may be obtained from the following formula:

$$a = \frac{2.6500 - 0.9984}{2.6500} \times \frac{G}{G - 0.9984}$$

For hydrometer *B*, the value of *a* for an assumed density of 1.0000 at 67 F. (19.4 C.) may be obtained from the following formula:

$$a = \frac{2.6500 - 1.0000}{2.6500} \times \frac{G}{G - 1.0000}$$

The values of *a* given by the two equations for different values of the specific gravity of soil *G*, are the same to two decimal places, and are as follows:

Specific Gravity, *G*	Constant, *a*
2.95	0.94
2.85	0.96
2.75	0.98
2.65	1.00
2.55	1.02
2.45	1.05
2.35	1.08

It is sufficiently accurate for ordinary tests to select the constant for the specific gravity nearest to that of the particular soil tested.

Example.—Assume that the weight of dispersed soil corrected for hygroscopic moisture is 48.5 g., the uncorrected reading of hydrometer *A* is 34, the specific gravity 2.41, and the temperature of suspension 70 F. (21 C.). The temperature correction is then +0.4 (Fig. 6 (*a*)) and the corrected reading, *R*, is 34 + 0.4 = 34.4. The specific gravity correction, *a*, is 1.05. Then:

$$P = \frac{34.4 \times 1.05}{48.5} \times 100 = 74.5.$$

(*d*) The percentage of the total test sample, including the fraction retained on the No. 10 (2000-micron) sieve, shall be obtained by multiplying the percentage of originally dispersed soil remaining in suspension by the expression:

$$\frac{100 - \text{percentage retained on No. 10 sieve}}{100}$$

Diameter of Soil Particles in Suspension

11. (*a*) The maximum diameter of the particles in suspension, corresponding to the percentage indicated by a given hydrometer reading, shall be as indicated by Stokes' law.

TABLE I.—MAXIMUM GRAIN DIAMETERS IN SUSPENSION UNDER ASSUMED CONDITIONS.

Time, min.	Maximum Grain Diameter in Suspension, mm.
1	0.078
2	0.055
5	0.035
15	0.020
30	0.014
60	0.010
250	0.005
1440	0.002

According to Stokes' law:

$$d = \sqrt{\frac{30nL}{980\,(G - G_1)\,T}}$$

where:

d = maximum grain diameter in millimeters,
n = coefficient of viscosity of the suspending medium (in this case water) in poises. Varies with changes in temperature of the suspending medium.
L = distance in centimeters through which soil particles settle in a given period of time,
T = time in minutes, period of sedimentation,
G = specific gravity of soil particles,
G_1 = specific gravity of the suspending medium (in this case G_1 = 0.9984, or approximately 1.0).

(*b*) The maximum grain diameters in suspension for assumed conditions and corresponding to the periods of sedimentation specified in this procedure are given in Table I. These grain diameters

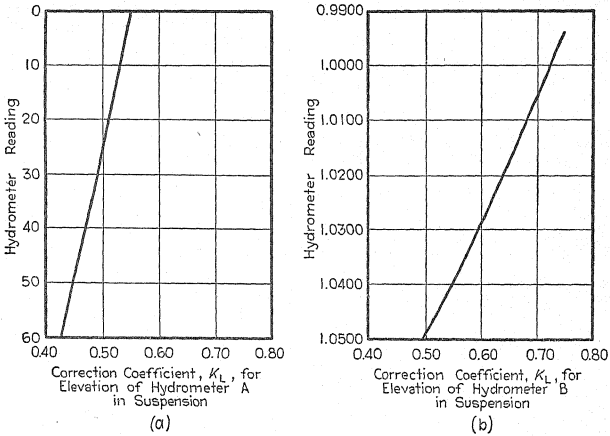


FIG. 7.—Correction Coefficient Curves for Elevation of Hydrometer.

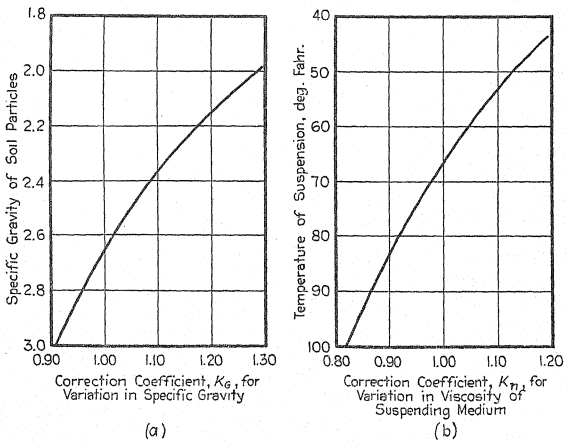


FIG. 8.—Correction Coefficient Curves for Specific Gravity and Viscosity.

shall be corrected for the conditions of the test by applying the proper correction factors as described and explained below.

(*c*) The grain diameters given in Table I are calculated according to the following assumptions:

L, the distance through which the particles fall, is constant and equal to 32.5 cm.

n, the coefficient of viscosity, equals 0.0102, that of water at 67 F. (19.4 C.).[4]

G, the specific gravity of the soil particles, is constant and equal to 2.65.

NOTE.—As a matter of fact, the hydrometer reading is dependent, not on the particles distributed throughout a depth of 32.5 cm. in the suspension, but on those existing in that portion of the suspension holding the hydrometer.

(*d*) In order to use Stokes' law to determine the diameter of the particles it is necessary to know the distance through which these particles fall in a given time. Since the density throughout a suspension is not uniform and varies with the grading of the material in suspension and the time of sedimentation, a fixed distance cannot be used. For hydrometer *B* the depth of the center of volume of the hydrometer below the surface of the suspension can be taken as the distance through which the particles may be assumed to fall. In the case of hydrometer *A*, it has been found by experiment that for the methods of dispersion described in this procedure an assumed distance which bears a constant ratio to the depth of the hydrometer in the suspension, but which is less than the distance indicated by the center of volume of the hydrometer, gives closer agreement to mechanical analysis performed by the pipette methods. The assumed distance of fall is taken as 0.42 of the distance from the surface of the suspension to the elevation of the bottom of hydrometer *A*.

(*e*) The specific gravity of the soil particles and the temperature of the suspension are likely to vary from those assumed in the preparation of Table I. A better approximation of the true diameters of the soil particles is obtained by applying correction coefficients to the values given by Table I.

(*f*) Curves from which these coefficients may be derived are given in Figs. 7 and 8. The correction coefficients for elevation of hydrometer (Fig. 7 (*a*) and (*b*)) are obtained experimentally for each hydrometer in use. The coefficients for the specific gravity and the viscosity correction (Fig. 8 (*a*) and (*b*), respectively) are independent of the apparatus used in the test.

(*g*) Multiplication by the coefficients shown in Fig. 7 (*a*) and (*b*) gives the maximum grain size at the reference elevation of hydrometers *A* and *B*, respectively, instead of that at a depth of 32.5 cm. These coefficients vary with the hydrometer reading and may be obtained from the following formula:

$$K_L = \sqrt{\frac{\text{assumed depth of fall in cm.}}{32.5}}$$

(*h*) Multiplication by the coefficients shown in Fig. 8 (*a*) corrects for variation in specific gravity from that on which the sizes given in Table I are based. These coefficients may be obtained from the following formula:

$$K_a = \sqrt{\frac{1.65}{\text{sp. gr. of soil particle} - 1}}$$

(*i*) Multiplication by the coefficients shown in Fig. 8 (*b*) corrects for the viscosity of water at temperatures other than 67 F. (19.4 C.), the temperature of the suspension assumed in the preparation of Table I. The viscosity correc-

[4] *Smithsonian Physical Tables*, 7th revised edition, 1921, p. 155.

tion coefficient may be obtained from the following formula:

$$K_n = \sqrt{\frac{n_1}{0.0102}}$$

where n_1 = viscosity coefficient at given temperature.

Example.—After a period of sedimentation of 1 min. the grain diameter indicated by Table I is 0.078 mm. Assume that the uncorrected reading of hydrometer *A* is 34, the specific gravity is 2.41, and the temperature of the suspension is 70 F. (21 C.). The correction coefficients in this case are 0.48, corresponding to a hydrometer reading of 34 (Fig. 7 (*a*)); 1.08, corresponding to a specific gravity of 2.41 (Fig. 8 (*a*)); and 0.98, corresponding to a water temperature of 70 F. (21 C.) (Fig. 8 (*b*)). The corrected grain diameter then becomes:

d = 0.078 mm. × 0.48 × 1.08 × 0.98 = 0.040 mm.

Sieve Analysis

12. (*a*) The percentage of the dispersed soil sample retained on each of the sieves in the sieve analysis shall be obtained by dividing the weight of fraction retained on each sieve by the oven-dry weight of the dispersed soil and multiplying by 100.

(*b*) The percentages of the total test sample, including the fraction retained on the No. 10 (2000-micron) sieve, shall be obtained by multiplying these values by the expression:

$$\frac{100 - \text{percentage retained on No. 10 sieve}}{100}$$

Plotting

13. The percentages of grains of different grain diameters shall be plotted on semilogarithmic paper to obtain a "soil grain diameter accumulation curve." A curve of this character is shown in Fig. 9.

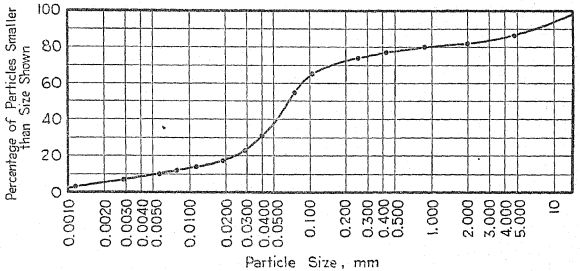


FIG. 9.—Grain Size Accumulation Curve, Soil Sample No. 4422X.

Report

14. The results, read from the accumulation curve, shall be reported as follows:

(*a*) Percentage of particles larger than 2 mm.

(*b*) Percentage of coarse sand, 2.0 to 0.25 mm.

(*c*) Percentage of fine sand, 0.25 to 0.05 mm.

(*d*) Percentage of silt, 0.05 to 0.005 mm.

(*e*) Percentage of clay, smaller than 0.005 mm.

(*f*) Percentage of colloids, smaller than 0.001 mm.

Standard Method of Test for

LIQUID LIMIT OF SOILS[1]

A.S.T.M. Designation: D 423 - 39

ADOPTED, 1939.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 423; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Liquid Limit

1. The liquid limit of a soil is that moisture content, expressed as a percentage of the weight of the oven-dried soil, at which the soil will just begin to flow when lightly jarred ten times.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Evaporating Dish.*—A porcelain evaporating dish about 4½ in. in diameter.

(*b*) *Spatula.*—A spatula or pill knife having a blade about 3 in. in length and about ¾ in. in width.

(*c*) *Liquid Limit Device.*—A mechanical device consisting of a brass dish and carriage, constructed according to the plan and dimensions shown in Fig. 1.

(*d*) *Grooving Tool.*—A combined grooving tool and gage conforming to the dimensions shown in Fig. 1.

(*e*) *Containers.*—Suitable containers such as matched watch glasses which will prevent loss of moisture during weighing.

(*f*) *Balance.*—A balance sensitive to 0.1 g.

HAND METHOD

Sample

3. A sample weighing about 30 g. shall be taken from the thoroughly mixed portion of the material passing the No. 40 (420-micron) sieve which has been obtained in accordance with the Standard Method of Preparing Soil Samples for Mechanical Analysis and Determination of Subgrade Soil Constants (A.S.T.M. Designation: D 421) of the American Society for Testing Materials.[3]

Procedure

4. (*a*) The air-dried soil shall be placed in the evaporating dish and thoroughly mixed with distilled water until the mass becomes a thick paste. The mass of soil shall then be shaped into a small layer about ⅜ in. in thickness at the center and divided into two portions with the grooving tool, as shown in the illustration at the top of Fig. 2.

(*b*) The dish shall be held firmly in

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-18 on Soils for Engineering Purposes.

[2] Prior to adoption as standard, this method was published as tentative from 1936 to 1939, being revised in 1938.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

one hand, with the groove parallel to the line of sight, and tapped lightly with a horizontal motion against the heel of the other hand ten times. The intensity of the blows shall be such that the effect on the soil sample is equivalent to that produced by 25 shocks applied to a sample of the soil at the same moisture content by dropping the brass cup of the mechanical device through a distance of the liquid limit, and dry soil shall be added and the procedure repeated.

(*d*) When the lower edges of the two portions of the soil cake just flow together, as shown in the illustration at the bottom of Fig. 2, after ten blows have been struck, the moisture content equals the liquid limit. To determine definitely whether the two portions are actually joined, the spatula may be used to push

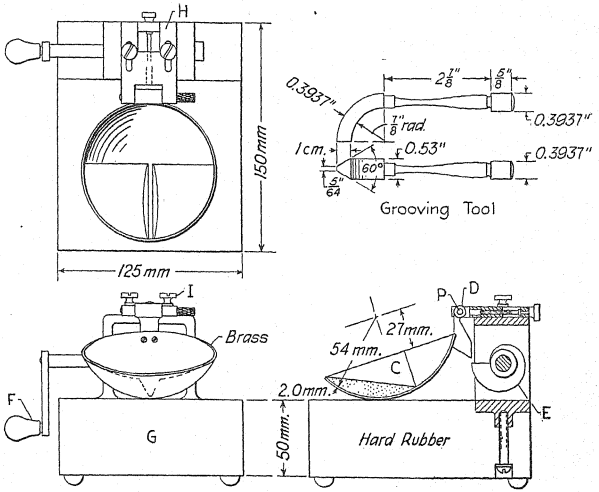


FIG. 1.—Liquid Limit Device.

1 cm. (0.3937 in.) at the rate of two drops per second.

(*c*) If the lower edges of the two soil portions do not flow together, as shown in the illustration at the bottom of Fig. 2, after ten blows have been struck, the moisture content is below the liquid limit. More water shall be added and the procedure repeated. If the lower edges meet before ten blows have been struck, the moisture content is above one away from the other. If the two portions separate along the original line of division, the end point has not been reached, and the procedure shall be repeated with the addition of a small amount of water.

(*e*) A small quantity of soil from that portion of the soil cake which was flowed shall be removed and placed in a pair of watch glasses or other suitable containers. The container and soil shall

then be weighed and the weight recorded. The soil in the container shall be oven-dried to constant weight at 110 C., and weighed. This weight shall be recorded and the loss in weight due to drying shall be recorded as the weight of water.

Calculation

5. The liquid limit is expressed as the moisture content in percentage of the weight of the oven-dried soil and shall be calculated as follows:

$$\text{Liquid limit} = \frac{\text{wt. of water}}{\text{wt. of oven-dry soil}} \times 100$$

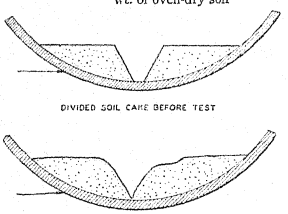


FIG. 2.—Diagram Illustrating Liquid Limit Test.

MECHANICAL METHOD

Calibration of Mechanical Device

6. By means of the gage attached to the grooving tool, and the adjustment plate, *H*, Fig. 1, the height to which the cup, *C*, is lifted shall be adjusted so that the point on the cup which comes in contact with the base is exactly 1 cm. (0.3937 in.) above the base. The adjustment plate, *H*, shall then be secured by tightening the screws, *I*.

Procedure

7. (*a*) A sample weighing about 100 g. shall be taken from the thoroughly mixed portion of the air-dried soil passing the No. 40 (420-micron) sieve which has been obtained in accordance with the Standard Method of Preparing Soil Samples for Mechanical Analysis and Determination of Subgrade Soil Constants (A.S.T.M. Designation: D 421) of the American Society for Testing Materials.[3] This sample shall be placed in the evaporating dish and thoroughly mixed with a measured quantity of distilled water to a putty-like consistency. A portion of this sample shall then be placed in the brass cup in the position shown in Fig. 1, leveled off to a depth of 1 cm., and divided by means of the grooving tool along the diameter through the centerline of the cam follower.

(*b*) The cup shall then be attached to the carriage and by turning the crank, *F*, at the rate of two rotations per second lifted and dropped until the two sides of the sample come into contact at the bottom of the groove along a distance of about ½ in. The number of shocks shall be recorded. The moisture content of the soil shall then be determined on a portion taken from around the groove, by the method previously described in Section 4 in connection with the hand method.

(*c*) The foregoing operations shall be repeated for two additional portions of the sample to which sufficient water has been added to bring the soil to a more fluid condition. The object of this procedure is to obtain samples of such consistency that the number of shocks required to close the groove will be below and above 25.

Preparation of Flow Curve

8. A "flow curve" representing the relation between moisture contents and corresponding numbers of shocks shall be plotted on a semilogarithmic graph with the moisture contents as abscissae on the arithmetical scale, and the numbers of shocks as ordinates on the logarithmic scale.

Liquid Limit

9. The moisture content corresponding to the intersection of the flow curve with the 25 shock ordinate shall be taken as the liquid limit of the soil.

Standard Method of Test for

PLASTIC LIMIT AND PLASTICITY INDEX OF SOILS[1]

A.S.T.M. Designation: D 424 - 39

ADOPTED, 1939.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 424; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Plastic Limit

1. The plastic limit of a soil is the lowest moisture content, expressed as a percentage of the weight of the oven-dried soil, at which the soil can be rolled into threads $\frac{1}{8}$ in. in diameter without the threads breaking into pieces.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Evaporating Dish.*—A porcelain evaporating dish about $4\frac{1}{2}$ in. in diameter.

(*b*) *Spatula.*—A spatula or pill knife having a blade about 3 in. in length and about $\frac{3}{4}$ in. in width.

(*c*) *Surface for Rolling.*—A ground-glass plate or piece of rough-textured paper on which to roll the sample.

(*d*) *Containers.*—Suitable containers, such as matched watch glasses, which will prevent loss of moisture during weighing.

(*e*) *Balance.*—A balance sensitive to 0.1 g.

Sample

3. A sample weighing about 15 g. shall be taken from the thoroughly mixed portion of the material passing the No. 40 (420-micron) sieve, which has been obtained in accordance with the Standard Method of Preparing Soil Samples for Mechanical Analysis and Determination of Subgrade Soil Constants (A.S.T.M. Designation: D 421) of the American Society for Testing Materials.[3]

Procedure

4. The air-dried soil shall be placed in the evaporating dish and mixed with distilled water until the mass becomes plastic enough to be easily shaped into a ball. The ball of soil shall then be rolled between the palm of the hand and the ground-glass plate or piece of rough-textured paper with just sufficient pressure to form the soil mass into a thread (Fig. 1 (*a*)). When the diameter of the resulting thread becomes $\frac{1}{8}$ in. the soil shall be kneaded together and again rolled out. This process shall be con-

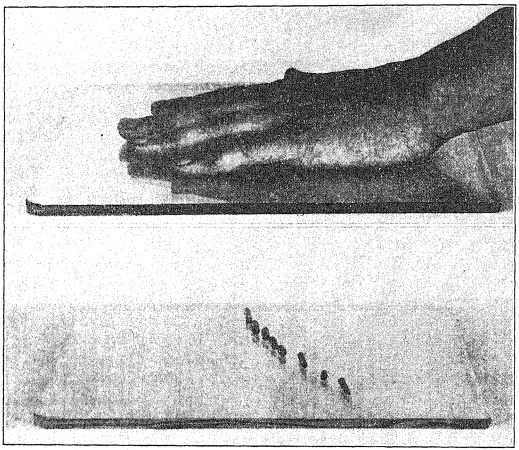
[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-18 on Soils for Engineering Purposes.

[2] Prior to adoption as standard, this method was published as tentative from 1935 to 1939, being revised in 1938.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

tinued until the crumbling of the soil (as shown in Fig. 1 (*b*)) prevents the formation of the thread. The portions of the crumbled soil shall then be gathered together and placed in watch glasses or other suitable containers. The container and soil shall be weighed and the weight recorded. The soil in the container shall then be oven-dried to constant weight at 110 C., and weighed. This weight shall be recorded and the loss in weight shall be recorded as the weight of water.

(a) Rolling of soil threads in plastic limit test.



(b) Crumbled soil threads resulting from plastic limit test.

FIG. 1.—Plastic Limit Test.

Calculation

5. (*a*) *Plastic Limit.*—The plastic limit is expressed as the moisture content in percentage of the weight of the oven-dry soil and shall be calculated as follows:

$$\text{Plastic limit} = \frac{\text{wt. of water}}{\text{wt. of oven-dry soil}} \times 100$$

(*b*) *Plasticity Index.*—The plasticity index of a soil is the difference between its liquid limit and its plastic limit and shall be calculated as follows:

$$\text{Plasticity index} = \text{liquid limit} - \text{plastic limit}$$

NOTE.—Liquid limit shall be determined in accordance with the Standard Method of Test for Liquid Limit of Soils (A.S.T.M. Designation: D 423) of the American Society for Testing Materials.[3]

Standard Method of Test for

CENTRIFUGE MOISTURE EQUIVALENT OF SOILS[1]

A.S.T.M. Designation: D 425 – 39

ADOPTED, 1939.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 425; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Centrifuge Moisture Equivalent

1. The centrifuge moisture equivalent of a soil is the amount of moisture, expressed as a percentage of the weight of the oven-dried soil, retained by the soil which has been first saturated with water and then subjected to a force equal to 1000 times the force of gravity for 1 hr.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Gooch Crucible.*—A porcelain Gooch crucible with perforated bottom. The crucible shall be about $1\frac{1}{2}$ in. in height and the diameter shall be about 1 in. at the top and $\frac{3}{4}$ in. at the bottom, outside dimensions.

(*b*) *Filter Paper.*—A circular piece of filter paper just large enough to cover the inside bottom of the Gooch crucible.

(*c*) *Trunnion Cup.*—A Babcock trunnion cup fitted with a brass cap and with a suitable device for supporting the Gooch crucible $\frac{1}{2}$ in. above the bottom of the cup in such a manner that the water ejected during the centrifuging operation shall not come in contact with the crucible and contents and, furthermore, that air may circulate freely about the crucible within the cup. Suitable fittings for the Babcock cup are shown in Fig. 1.

(*d*) *Centrifuge.*—The centrifuge shall be of such size and so driven that a force equal to 1000 times the force of gravity may be exerted on the center of gravity of the soil sample.

(*e*) *Balance.*—A balance sensitive to 0.1 g.

Sample

3. A 5-g. sample shall be taken from the thoroughly mixed portion of the material passing the No. 40 (420-micron) sieve which has been obtained in accordance with the Standard Method of Preparing Soil Samples for Mechanical Analysis and Determination of Subgrade Soil Constants (A.S.T.M. Designation: D 421) of the American Society for Testing Materials.[3]

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-18 on Soils for Engineering Purposes.

[2] Prior to adoption as standard, this method was published as tentative from 1935 to 1939, being revised in 1938.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Number of Tests

4. Tests shall be made in duplicate.

Procedure

5. (*a*) The sample shall be placed in the Gooch crucible, in which has previously been placed a piece of wet filter paper which just covers the bottom of the crucible. The crucible shall be placed in a pan of distilled water and the sample allowed to take up moisture until completely saturated, as indicated by the presence of free water on the surface of the sample. It shall then be placed in a humidifier for at least 12 hr. to insure uniform distribution of moisture throughout the soil mass. All free water then remaining on the surface of the sample shall be poured off, and the crucible placed in a Babcock trunnion cup fitted as described in Section 2 (*c*).

(*b*) The sample shall be centrifuged for a period of 1 hr. at a speed which, for the diameter of head used, will exert a centrifugal force 1000 times the force of gravity upon the center of gravity of the soil sample. Immediately after centrifuging, the crucible and contents shall be weighed and the weight recorded as the weight of crucible and contents after centrifuging. The sample shall then be oven-dried to constant weight at 110 C., and weighed. This weight shall be recorded as the weight of crucible and contents after drying.

(*c*) *Waterlogging.*—When free water is observed on the top of the sample after the centrifuging operation, the soil is said to have waterlogged. This water shall not be removed but shall be weighed with the sample.

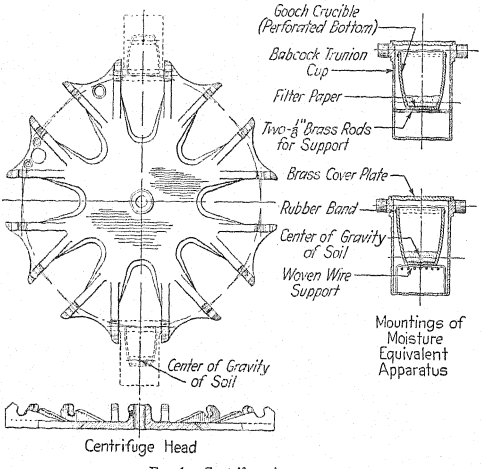


FIG. 1.—Centrifuge Apparatus.

Calculation

6. The centrifuge moisture equivalent of the soil shall be calculated as follows:

$$M_e = \frac{(A - b) - (A_1 - b_1)}{A_1 - (c + b_1)} \times 100$$

where:

M_e = centrifuge moisture equivalent,
A = weight of crucible and contents after centrifuging,
A_1 = weight of crucible and contents after drying,
c = weight of crucible,
b = weight of wet filter paper, and
b_1 = weight of dry filter paper.

Reproducibility of Results

7. The variation between the two values obtained in the duplicate tests should not exceed 1 per cent for values of the moisture equivalent up to 15 and 2 per cent for values above 15.

Standard Method of Test for

FIELD MOISTURE EQUIVALENT OF SOILS[1]

A.S.T.M. Designation: D 426 - 39

ADOPTED, 1939.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 426; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Field Moisture Equivalent

1. The field moisture equivalent of a soil is defined as the minimum moisture content, expressed as a percentage of the weight of the oven-dried soil, at which a drop of water placed on a smoothed surface of the soil will not immediately be absorbed by the soil but will spread out over the surface and give it a shiny appearance.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Evaporating Dish.*—A porcelain evaporating dish about 4½ in. in diameter.

(*b*) *Spatula.*—A spatula or pill knife having a blade about 3 in. in length and about ¾ in. in width.

(*c*) *Pipette.*—A pipette, burette, or similar device for adding water dropwise.

(*d*) *Containers.*—Suitable containers such as matched watch glasses which will prevent loss of moisture during weighing.

(*e*) *Balance.*—A balance sensitive to 0.1 g.

Sample

3. A sample weighing about 50 g. shall be taken from the thoroughly mixed portion of the material passing the No. 40 (420-micron) sieve, which has been obtained in accordance with the Standard Method of Preparing Soil Samples for Mechanical Analysis and Determination of Subgrade Soil Constants (A.S.T.M. Designation: D 421) of the American Society for Testing Materials.[3]

Procedure

4. The air-dried sample shall be placed in the evaporating dish and mixed with distilled water. Distilled water shall be added in small amounts and the sample thoroughly mixed after each addition of water. When the wetted soil forms into balls under manipulation, the sample shall be smoothed with a light stroke of the spatula and a drop of water placed on the smoothed surface. If the drop of water disappears in 30 sec. a

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-18 on Soils for Engineering Purposes.

[2] Prior to adoption as standard, this method was published as tentative from 1935 to 1939, being revised in 1938.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

small increment of water (a few drops) shall be mixed with the sample, and the procedure repeated until the drop of water does not disappear in 30 sec. but spreads over the smoothed surface leaving a shiny appearance. A small portion of the soil on which the last drop of water was placed shall then be removed and placed between two watch glasses or other suitable containers. The weight of the container and wet soil shall be determined and recorded. The sample shall then be oven-dried to constant weight at 110 C., and weighed. This weight shall be recorded and the difference in weight shall be recorded as the weight of water.

Calculation

5. The field moisture equivalent shall be calculated as follows:

$$\text{Field moisture equivalent} = \frac{\text{wt. of water}}{\text{wt. of oven-dried soil}} \times 100$$

Standard Method of Test for

SHRINKAGE FACTORS OF SOILS[1]

A.S.T.M. Designation: D 427 - 39

ADOPTED, 1939.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 427; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test is intended for obtaining the data from which the following subgrade soil constants may be calculated:

Shrinkage limit,
Shrinkage ratio,
Volumetric change,
Lineal shrinkage, and
Specific gravity (approximate).

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Evaporating Dish.*—A porcelain evaporating dish about $4\frac{1}{2}$ in. in diameter.

(*b*) *Spatula.*—A spatula or pill knife having a blade about 3 in. in length and about $\frac{3}{4}$ in. in width.

(*c*) *Shrinkage Dish.*—A circular porcelain or monel metal milk dish having a flat bottom and being about $1\frac{3}{4}$ in. in diameter and about $\frac{1}{2}$ in. in height.

(*d*) *Straightedge.*—A steel straightedge about 12 in. in length.

(*e*) *Glass Cup.*—A glass cup about 2 in. in diameter and about 1 in. in height, the top rim of which is ground smooth and level.

(*f*) *Glass Plate.*—A glass plate with three metal prongs for immersing the soil pat in mercury, as shown in Fig. 1.

(*g*) *Graduate.*—A glass graduate having a capacity of 25 ml. and graduated to 0.2 ml.

(*h*) *Balance.*—A balance sensitive to 0.1 g.

(*i*) *Mercury.*—Sufficient mercury to fill the glass cup to overflowing.

Sample

3. A sample weighing about 30 g. shall be taken from the thoroughly mixed portion of the material passing the No. 40 (420-micron) sieve which has been obtained in accordance with the Standard Method of Preparing Soil Samples for Mechanical Analysis and Determination of Subgrade Soil Constants (A.S.T.M. Designation: D 421) of the American Society for Testing Materials.[3]

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-18 on Soils for Engineering Purposes.

[2] Prior to adoption as standard, this method was published as tentative from 1935 to 1939, being revised in 1938.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Procedure

4. (*a*) The sample shall be placed in the evaporating dish and thoroughly mixed with distilled water in amount sufficient to fill the soil voids completely and to make the soil pasty enough to be readily worked into the shrinkage dish without the inclusion of air bubbles. The amount of water required to furnish friable soils with the desired consistency is equal to or slightly greater than the liquid limit, and the amount necessary to furnish plastic soils with the desired consistency

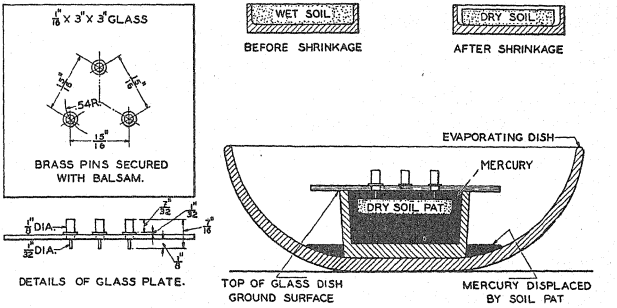


FIG. 1.—Apparatus for Determining the Volumetric Change of Subgrade Soils.

dish tapped until the soil is thoroughly compacted and all included air has been brought to the surface. More soil shall be added and the tapping shall be continued until the dish is completely filled and excess soil stands out about its edge. The excess soil shall then be struck off with a straightedge, and all soil adhering to the outside of the dish shall be wiped off.

(*c*) The dish when filled and struck off level shall be weighed immediately and the weight recorded as the weight of dish and wet soil. The soil pat shall be allowed to dry in air until the color of the pat turns from dark to light. It shall then be oven-dried to constant weight at 110 C. and weighed, the weight being recorded as the weight of dish and dry soil. The weight of the empty dish shall be determined and recorded. The capacity of the dish in cubic centimeters, which is also the volume of the wet soil pat, shall be determined by filling the dish to overflowing with mercury, removing the excess by pressing a glass plate firmly over the top of the dish, and measuring by means of a glass graduate

may exceed the liquid limit by as much as 10 per cent.

(*b*) The inside of the shrinkage dish shall be coated with a thin layer of vaseline or some other heavy grease to prevent the adhesion of the soil to the dish. An amount of the wetted soil equal to about one-third the volume of the dish shall be placed in the center of the dish, and the soil caused to flow to the edges by tapping the dish on a firm surface cushioned by several layers of blotting paper or similar material. An amount of soil shall be added approximately equal to the first portion, and the

the volume of mercury held in the dish. This volume shall be recorded as the volume of the wet soil pat, V.

(*d*) The volume of the dry soil pat shall be determined by removing the pat from the shrinkage dish and immersing it in the glass cup full of mercury in the following manner: The glass cup shall be filled to overflowing with mercury and the excess mercury shall be removed by pressing the glass plate with the three prongs (Fig. 1) firmly over the top of the cup. Any mercury which may be adhering to the outside of the cup shall be carefully wiped off. The cup, filled with mercury, shall be placed in the evaporating dish, and the soil pat shall be placed on the surface of the mercury. It shall then be carefully forced under the mercury by means of the glass plate with the three prongs (Fig. 1) and the plate shall be pressed firmly over the top of the cup. It is essential that no air be trapped under the soil pat. The volume of the mercury so displaced shall be measured in the glass graduate and recorded as the volume of the dry soil pat, V_o.

Calculation of Moisture Content

5. The moisture content of the soil at the time it was placed in the dish expressed as a percentage of the dry weight of the soil shall be calculated as follows:

$$M = \frac{W - W_0}{W_0} \times 100$$

where:

M = moisture content of the soil when placed in the dish,
W = weight of wet soil pat obtained by subtracting the weight of the shrinkage dish from the weight of the dish and wet pat, and
W_o = weight of dry soil pat obtained by subtracting the weight of the shrinkage dish from the weight of the dish and dry pat.

Shrinkage Limit

6. (*a*) The shrinkage limit of a soil is that moisture content, expressed as a percentage of the weight of the oven-dried soil, at which a reduction in moisture content will not cause a decrease in the volume of the soil mass, but at which an increase in moisture content will cause an increase in the volume of the soil mass.

(*b*) The shrinkage limit, S, shall be calculated from the data obtained in the volumetric shrinkage determination by the following formula:

$$S = M - \left(\frac{V - V_0}{W_0} \times 100 \right)$$

where:

S = shrinkage limit
M = moisture content of wet soil, in percentage of the weight of oven-dried soil,
V = volume of wet soil pat,
V_o = volume of dry soil pat, and
W_o = weight of oven-dried soil pat.

(*c*) *Optional Method.*—When both the true specific gravity, G, and the shrinkage ratio, R, are known, the shrinkage limit may be calculated as follows:

$$S = \left(\frac{1}{R} - \frac{1}{G} \right) \times 100$$

Shrinkage Ratio

7. (*a*) The shrinkage ratio of a soil is the ratio between a given volume change, expressed as a percentage of the dry volume, and the corresponding change in moisture content above the shrinkage limit, expressed as a percentage of the weight of the oven-dried

soil. It equals the apparent specific gravity of the dried soil pat.

(*b*) The shrinkage ratio, *R*, shall be calculated from the data obtained in the volumetric shrinkage determination by the following formula:

$$R = \frac{W_0}{V_0}$$

Volumetric Change

8. (*a*) The volumetric change of a soil for a given moisture content is the volume change, expressed as a percentage of the dry volume, of the soil mass when the moisture content is reduced from the stipulated percentage to the shrinkage limit. This stipulated moisture content is usually taken as the field moisture equivalent.

(*b*) The volumetric change shall be calculated from the data obtained in the volumetric shrinkage determination by the following formula:

$$\text{Volumetric change} = (M_1 - S)R$$

where M_1 = given moisture content.

If, as is customary, the volumetric change from the field moisture equivalent is desired, the formula assumes the following form:

$$\text{Volumetric change, } C_f = \text{volumetric change from field moisture equivalent}$$

$$\text{Volumetric change, } C_f = (F - S)R$$

where F = field moisture equivalent.

Lineal Shrinkage

9. (*a*) The lineal shrinkage of a soil for a given moisture content is the decrease in one dimension, expressed as a percentage of the original dimension, of the soil mass when the moisture content is reduced from an amount equal to the field moisture equivalent to the shrinkage limit.

(*b*) The lineal shrinkage shall be obtained either by means of the following formula or by means of the curve

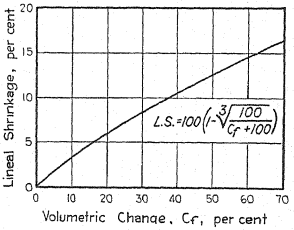


FIG. 2.—Curve for Determining Lineal Shrinkage.

shown in Fig. 2, which represents this relation:

$$\text{Lineal shrinkage} = 100\left(1 - \sqrt[3]{\frac{100}{C_f + 100}}\right)$$

Specific Gravity

10. (*a*) The specific gravity of a soil is the weight of the oven-dried soil divided by the true volume of the soil particles.

(*b*) The specific gravity may be calculated from the data obtained in the volumetric shrinkage test by the following formula:

$$\text{Sp. gr.} = \frac{1}{\frac{1}{R} - \frac{S}{100}}$$

Standard Method of Test for

MOISTURE-DENSITY RELATIONS OF SOIL-CEMENT MIXTURES[1]

A.S.T.M. Designation: D 558 - 44

ADOPTED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 558; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test is intended for determining the relationship between the moisture content of soil-cement mixtures and resulting densities (oven-dry weight per cubic foot) when the soil-cement mixture is compacted in the laboratory, before cement hydration, as specified herein.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Mold.*—A cylindrical metal mold having a capacity of $\frac{1}{30}$ cu. ft. with an internal diameter of 4.0 in. and a height of approximately 4.6 in., which has a detachable collar assembly approximately $2\frac{1}{2}$ in. in height, to permit preparing compacted specimens of soil-cement mixtures 4.0 in. in diameter and approximately 4.6 in. in height which have a volume of $\frac{1}{30}$ cu. ft. The mold and collar shall be fastened to a detachable base. (See Fig. 1.)

(*b*) *Rammer.*—A metal rammer having a 2-in. diameter circular face and weighing 5.5 lb. The rammer shall be equipped with a suitable arrangement to control the specified drop.

(*c*) *Sleeve.*—Closed cylindrical sleeve slightly less than 4.0 in. in diameter or similar device for removing compacted specimens from the mold.

(*d*) *Balances.*—A balance or scale of 25-lb. capacity sensitive to 0.01 lb.; and a 100-g. capacity balance sensitive to 0.1 g.

(*e*) *Drying Oven.*—A thermostatically controlled drying oven capable of maintaining temperatures of about 110 C. (230 F.) for drying moisture samples.

Procedure

3. (*a*) The air-dry soil first shall be pulverized to pass a No. 4 (4760-micron) sieve so as to separate the soil particles without reducing the particle size.

(*b*) The required cement shall be added to the pulverized soil. The test shall be performed only on that portion of the soil-cement mixture which will

[1] Under the standardization procedure of the Society, this method is under the joint jurisdiction of A.S.T.M. Committee D-4 on Road and Paving Materials and Committee D-18 on Soils for Engineering Purposes.

[2] Prior to adoption as standard, this method was published as tentative from 1940 to 1944.

pass a No. 4 (4760-micron) sieve. A quantity of water sufficient to produce slight cohesion shall then be thoroughly mixed with the soil-cement sample to permit ready compaction. The thoroughly mixed soil-cement sample shall be immediately compacted in the mold in three equal layers, to give a total compacted depth of about 5 in.; each layer being compacted by 25 blows of the rammer dropping free from a height of 12 in. above the elevation of the soil when a sleeve-type rammer is used or 12

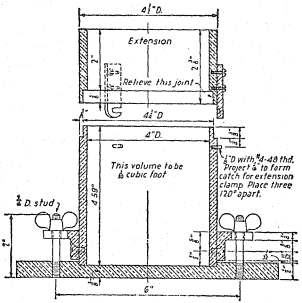


FIG. 1.—Cylindrical Mold for Moisture-Density Test.

in. above the approximate elevation of each finally compacted layer when a stationary mounted type rammer is used. During compaction, the mold shall rest on a uniform, rigid foundation weighing 200 lb. or its equivalent. The blows shall be uniformly distributed over the surface of the layer being compacted. After compacting, the collar shall be removed and the top carefully trimmed to the exact height of the mold with a steel straightedge to produce a specimen approximately 4.6 in. in height and having a volume of $\frac{1}{30}$ cu. ft.

(*c*) The weight of the compacted soil-cement mixture shall be determined, the material removed from the cylinder, sliced vertically in the center and a 100-g. sample taken from the center, weighed immediately, dried in an oven at 110 C. (230 F.) for at least 12 hr. or to constant weight. This procedure establishes the moisture-density relation of the initial soil-cement mixture.

(*d*) The soil-cement mixture shall be again finely pulverized, so as to separate the soil particles without reducing the particle size, to pass a No. 4 (4760-micron) sieve and a small increment of moisture carefully added and thoroughly mixed to insure uniform distribution. Then the moistened and pulverized material shall be compacted in the mold in three layers as described in Paragraph (*b*), weighed, and a moisture determination made to establish the moisture-density relation for a slightly moistened soil-cement mixture. The soil-cement mixture again shall be finely pulverized, so as to separate the soil particles without reducing the particle size, to pass a No. 4 sieve and additional increments of water added and the same procedure followed of mixing, compacting in the mold, weighing, and making moisture determinations until the moisture content of the soil-cement mixture reaches a condition where it is difficult to pulverize the moistened materials, or it has a moisture content near the liquid limit. This procedure will establish the moisture-density relations of a soil-cement mixture for the moisture contents practicable to handle in the field.

Calculation

4. The moisture content and oven-dry weight of the mixture as compacted shall

be calculated by means of the following formulas:

$$\text{Moisture, per cent} = \frac{\text{wt. of wet soil-cement} - \text{wt. of oven-dried soil-cement}}{\text{wt. of oven-dried soil-cement}} \times 100$$

$$\text{Dry weight (per cu. ft. of soil-cement as compacted)} = \frac{\text{wet wt. in lb. per cu. ft.}}{\text{percentage of moisture plus 100}} \times 100$$

Moisture-Density Relationship

5. (*a*) The calculations in Section 4 are made to determine the moisture content and corresponding compacted oven-dry weight (density) of the compacted soil-cement for each test made on the mixture. The oven-dry weights per cubic foot (density) of the soil shall be plotted as ordinates and the corresponding moisture contents as abscissas.

(*b*) *Optimum Moisture Content.*—When the moisture-density relations have been determined for a soil-cement mixture and the results plotted as indicated in Paragraph (*a*), it will be found that by connecting the plotted points with a smooth line a curve is produced which is, generally, parabolic in form. The moisture content producing the peak of the curve shall be termed the "optimum moisture content" of the soil-cement mixture under the above compaction.

(*c*) *Maximum Density.*—The oven-dry weight per cubic foot of moist soil-cement mixture at "optimum moisture content" shall be termed "maximum density" under the above compaction.

(*d*) When the curve resulting from connecting plotted points does not produce a parabolic-type curve with a definite peak, the "optimum moisture" and "maximum density" for such mixtures shall be determined from the results of additional tests conducted in conformance with the Standard Method of Wetting-and-Drying Test of Compacted Soil-Cement Mixtures (A.S.T.M. Designation: D 559)[3] and the Standard Method of Freezing-and-Thawing Test of Compacted Soil-Cement Mixtures (A.S.T.M. Designation: D 560)[3] on compacted soil-cement specimens. These specimens shall be molded with moisture contents approximately equal to the plastic limit of the soil and 1, 2, and 3 per cent above and below this governing moisture content. The densities of these specimens shall be those obtained with the prescribed moisture contents in the moisture-density test for the mixture. The cement content of these specimens shall be such that the soil-cement losses on the specimen giving the best results in these tests shall not exceed 10 per cent of the dry weight of the specimen. The moisture content and density corresponding to the specimen giving the best results shall be used as the "optimum moisture content" and "maximum density" of soil-cement mixtures which do not produce a parabolic-type curve.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Method of

WETTING-AND-DRYING TEST OF COMPACTED SOIL-CEMENT MIXTURES[1]

A.S.T.M. Designation: D 559 – 44

ADOPTED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 559; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test is intended for determining the soil-cement losses, moisture changes, and volume changes (swell and shrinkage) produced by repeated wetting and drying of compacted specimens of soil-cement mixtures of known composition and of known uniform density and moisture content.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Mold.*—A cylindrical metal mold having a capacity of $\frac{1}{30}$ cu. ft. with an internal diameter of 4.0 in. and a height of approximately 4.6 in., which has a detachable collar assembly approximately $2\frac{1}{2}$ in. in height, to permit preparing compacted specimens of soil-cement mixtures 4.0 in. in diameter and approximately 4.6 in. in height which have a volume of $\frac{1}{30}$ cu.ft. The mold and collar shall be fastened to a detachable base. (See Fig. 1.)

(*b*) *Rammer.*—A metal rammer having a 2-in. diameter circular face and weighing 5.5 lb. The rammer shall be equipped with a suitable arrangement to control the specified drop.

(*c*) *Sleeve.*—Closed cylindrical sleeve slightly less than 4.0 in. in diameter or similar device for removing compacted specimens from the mold.

(*d*) *Balances.*—A balance or scale of 25-lb. capacity sensitive to 0.01 lb.; and a 100-g. capacity balance sensitive to 0.1 g.

(*e*) *Drying Ovens.*—A thermostatically controlled drying oven capable of maintaining temperatures of about 110 C. (230 F.) for drying moisture samples.

A thermostatically controlled drying oven capable of maintaining temperatures of about 71 C. (160 F.) for drying compacted soil-cement specimens.

(*f*) *Moist Room.*—A moist room capable of maintaining a temperature of 21 ± 1.7 C. (70 ± 3 F.) and a relative humidity of not less than 90 per cent for seven-day storage of compacted specimens.

[1] Under the standardization procedure of the Society, this method is under the joint jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials and Committee D-18 on Soils for Engineering Purposes.

[2] Prior to adoption as standard, this method was published as tentative from 1940 to 1944.

(*g*) *Water Bath.*—Suitable tank for submerging compacted specimens in water at about 21 C. (70 F.).

(*h*) *Wire Scratch Brush.*—A wire scratch brush made of 2-in. by $\frac{1}{16}$-in. flat No. 26 gage wire bristles assembled in 50 groups of 10 bristles each and mounted to form five longitudinal rows and ten transverse rows of bristles on a $7\frac{1}{2}$-in. by $2\frac{1}{2}$-in. hardwood block.

Preparation of Material for Molding Specimens

3. This test shall be performed on soil-cement mixtures compacted in the mold, before cement hydration, to maximum density at optimum moisture content and in the same manner of compaction as specified in the Standard Method of Test for Moisture - Density Relations of Soil-Cement Mixtures (A.S.T.M. Designation: D 558) of the American Society for Testing Materials.[3] Representative roadway soil shall be used except as modified in the following Paragraph (*b*).

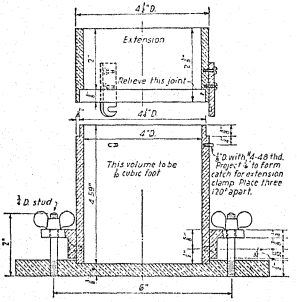


FIG. 1.—Cylindrical Mold for Molding Specimens.

(*a*) *For Soil-Cement Mixtures Containing No Material Retained on the No. 4 Sieve.*—A sufficient quantity of each soil-cement mixture to be investigated shall be prepared at optimum moisture content to provide two compacted specimens approximately 4.6 in. in height and 4.0 in. in diameter.

(*b*) *For Soil-Cement Mixtures Containing Material Retained on the No. 4 Sieve.*—When the soil-cement mixtures contain material retained on the No. 4 (4760-micron) sieve, the following procedures shall be followed which vary from that described in Paragraph (*a*): Since the specimens are 4 in. in diameter, it is necessary to prescribe the maximum size material to be used in the specimen and this size shall be set at $\frac{3}{4}$ in. Compensation shall be made for any material larger than $\frac{3}{4}$ in. by replacing it with an equal dry weight of No. 4 sieve to $\frac{3}{4}$-in. material of the same gradation as that contained in the original soil sample. A sufficient quantity of minus No. 4 sieve soil-cement material shall be prepared at the optimum moisture content to provide two compacted specimens 4.6 in. in height and 4.0 in. in diameter after the requisite amount of saturated, surface-moist, plus No. 4 sieve material, as defined above, has been added to the damp, minus No. 4 material.

Molding Specimens

4. (*a*) The prepared material shall be compacted in the mold in the same manner as that specified in the Standard Method of Test for Moisture - Density Relations of Soil-Cement Mixtures (A.S.T.M. Designation: D 558) of the American Society for Testing Materials,[3] with the further provision that before placing and compacting the next layer, smooth compaction planes produced

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

during compaction of each layer shall be scarified to form grooves, at right angles to each other, approximately $\frac{1}{2}$ in. in width and $\frac{1}{8}$ in. in depth and approximately $\frac{1}{4}$ in. apart. During compaction, a representative soil-cement mixture moisture sample shall be taken from the batch, weighed, dried in an oven at 110 C. (230 F.) for at least 12 hr. or to constant weight. The compacted specimens shall be removed from the mold, weighed, and the density determined; the volume shall be determined by (*1*) averaging the diameters measured to the nearest 0.02 in. (0.05 cm.) at right angles to each other at the center and the quarter points of the specimen and averaging the heights measured to the nearest 0.02 in. (0.05 cm.) at the quarter points on the circumference of the specimen, or (*2*) by immersion and displacement methods in a nonabsorbent liquid such as mercury.

(*b*) One specimen shall be identified on a metal tag (or other suitable device) as No. 1, together with other needed identification marks and shall be used to obtain data on moisture and volume changes during the test. The other specimen shall be identified as No. 2, together with other needed identification marks and shall be used to obtain data on soil-cement losses during this test.

(*c*) After the required data have been obtained, the specimens shall be placed on suitable carriers in the moist room and protected from free water for a period of seven days. The No. 1 specimens shall be weighed and measured daily to give data for calculating the moisture content and volume of the specimens each day.

Procedure

5. (*a*) At the end of the storage in the moist room, the specimens shall be submerged in tap water at room temperature for a period of 5 hr. and removed. The No. 1 specimen (volume and moisture change specimen) shall be weighed and measured.

(*b*) Both specimens shall be placed in an oven at about 71 C. (160 F.) for 42 hr. and removed. Then, both specimens shall be weighed and No. 1 specimen measured. Specimen No. 2 (soil-cement loss specimen) shall then be given two firm strokes on all areas with a wire scratch brush to remove all material loosened during wetting and drying. The specimen shall again be weighed after brushing. The moisture content of the material brushed from the specimen shall be determined directly, but if this is not possible, it may be assumed to be equal to the moisture content of the No. 1 specimen. The oven-dry (110 C., 230 F.) weight of material brushed from the specimen shall be calculated.

(*c*) The procedures described in Paragraphs (*a*) and (*b*) constitute one cycle (48 hr.) of wetting and drying. The specimens shall then be again submerged in water and the procedure continued. The data collected will permit calculations of volume and moisture changes of specimen No. 1 and the soil-cement losses of specimen No. 2.

(*d*) The wetting and drying of the No. 1 specimens shall be continued for 12 cycles or discontinued prior to 12 cycles should the measurements become inaccurate due to distortion or soil-cement loss of the specimens. The wetting and drying of the No. 2 specimens shall be continued for 12 cycles, if possible.

Calculation

6. The volume and moisture changes and the soil-cement losses of the specimens shall be calculated as follows:

(*a*) The difference between the volume of specimen No. 1 at the time of

molding and subsequent volumes shall be calculated as a percentage of the original volume.

(*b*) The moisture content of specimen No. 1 at the time of molding and subsequent moisture contents shall be calculated as a percentage of the original oven-dry weight of the specimen.

(*c*) The soil-cement loss of specimen No. 2 shall be calculated as a percentage of the original oven-dry weight of the specimen.

Standard Method of

FREEZING-AND-THAWING TEST OF COMPACTED SOIL-CEMENT MIXTURES[1]

A.S.T.M. Designation: D 560 – 44

ADOPTED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 560; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test is intended for determining the soil-cement losses, moisture changes, and volume changes (swell and shrinkage) produced by repeated freezing and thawing of compacted specimens of soil-cement mixtures of known composition and of known uniform density and moisture content.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Mold.*—A cylindrical metal mold having a capacity of $\frac{1}{30}$ cu.ft. with an internal diameter of 4.0 in. and a height of approximately 4.6 in., which has a detachable collar assembly approximately $2\frac{1}{2}$ in. in height, to permit preparing compacted specimens of soil-cement mixtures 4.0 in. in diameter and approximately 4.6 in. in height which have a volume of $\frac{1}{30}$ cu. ft. The mold and collar shall be fastened to a detachable base. (See Fig. 1.)

(*b*) *Rammer.*—A metal rammer having a 2-in. diameter circular face and weighing 5.5 lb. The rammer shall be equipped with a suitable arrangement to control the specified drop.

(*c*) *Sleeve.*—Closed cylindrical sleeve slightly less than 4.0 in. in diameter or similar device for removing compacted specimens from the mold.

(*d*) *Balances.*—A balance or scale of 25-lb. capacity sensitive to 0.01 lb.; and a 100-g. capacity balance sensitive to 0.1 g.

(*e*) *Drying Oven.*—A thermostatically controlled drying oven capable of maintaining temperatures of about 110 C. (230 F.) for drying moisture samples.

(*f*) *Freezing Cabinet.*—A freezing cabinet capable of maintaining temperatures of minus 23 C. (minus 10 F.) or lower.

(*g*) *Moist Room.*—A moist room capable of maintaining a temperature of 21 ± 1.7 C. (70 ± 3 F.) and a relative humidity of not less than 90 per cent for seven-day storage of compacted specimens and for thawing frozen specimens.

[1] Under the standardization procedure of the Society, this method is under the joint jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials and Committee D-18 on Soils for Engineering Purposes.

[2] Prior to adoption as standard, this method was published as tentative from 1940 to 1944.

(*h*) *Wire Scratch Brush.*—A wire scratch brush made of 2-in. by $\frac{1}{16}$-in. flat No. 26 gage wire bristles assembled in 50 groups of 10 bristles each and mounted to form five longitudinal rows and ten transverse rows of bristles on a $7\frac{1}{2}$-in. by $2\frac{1}{2}$-in. hardwood block.

Preparation of Material for Molding Specimens

3. This test shall be performed on soil-cement mixtures compacted in the mold, before cement hydration, to maximum density at optimum moisture content and in the same manner of compaction as specified in the Standard Method of Test for Moisture-Density Relations of Soil-Cement Mixtures (A.S.T.M. Designation: D 558) of the American Society for Testing Materials.[3] Representative roadway soil shall be used except as modified in the following Paragraph (*b*).

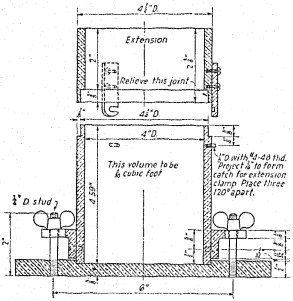


FIG. 1.—Cylindrical Mold for Molding Specimens.

(*a*) *For Soil-Cement Mixtures Containing No Material Retained on the No. 4 Sieve.*—A sufficient quantity of each soil-cement mixture to be investigated shall be prepared at optimum moisture content to provide two compacted specimens approximately 4.6 in. in height and 4.0 in. in diameter.

(*b*) *For Soil-Cement Mixtures Containing Material Retained on the No. 4 Sieve.*—When the soil-cement mixtures contain material retained on the No. 4 (4760-micron) sieve, the following procedures shall be followed which vary from that described in Paragraph (*a*): Since the specimens are 4 in. in diameter, it is necessary to prescribe the maximum size material to be used in the specimens and this size shall be set at $\frac{3}{4}$ in. Compensation shall be made for any material larger than $\frac{3}{4}$ in. by replacing it with an equal dry weight of No. 4 sieve to $\frac{3}{4}$-in. material of the same gradation as that contained in the original soil sample. A sufficient quantity of minus No. 4 sieve soil-cement material shall be prepared at optimum moisture content to provide two compacted specimens 4.6 in. in height and 4.0 in. in diameter after the requisite amount of saturated surface-moist, plus No. 4 sieve material, as defined above, has been added to the damp, minus No. 4 sieve material.

Molding Specimens

4. (*a*) The prepared material shall be compacted in the mold in the same manner as that specified in the Standard Method of Test for Moisture - Density Relations of Soil-Cement Mixtures (A.S.T.M. Designation: D 558) of the American Society for Testing Materials,[3] with the further provision that before placing and compacting the next layer, smooth compaction planes produced during compaction of each layer shall be scarified to form grooves, at right angles to each other, approximately $\frac{1}{8}$ in. in width and $\frac{1}{8}$ in. in depth and approximately $\frac{1}{4}$ in. apart. During compaction, a repre-

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

sentative soil-cement mixture moisture sample shall be taken from the batch, weighed, dried in an oven at 110 C. (230 F.) for at least 12 hr. or to constant weight. The compacted specimens shall be removed from the mold, weighed, and density determined; the volume shall be determined by (*1*) averaging the diameters measured to the nearest 0.02 in. (0.05 cm.) at right angles to each other at the center and the quarter points of the specimen and averaging the heights measured to the nearest 0.02 in. (0.05 cm.) at the quarter points on the circumference of the specimen, or (*2*) by immersion and displacement methods in a nonabsorbent liquid such as mercury.

(*b*) One specimen shall be identified on a metal tag (or other suitable device) as No. 1, together with other needed identification marks and shall be used to obtain data on moisture and volume changes during the test. The other specimen shall be identified as No. 2, together with other needed identification marks and shall be used to obtain data on soil-cement losses during this test.

(*c*) After the required data have been obtained, the specimens shall be placed on suitable carriers in the moist room and protected from free water for a period of seven days. The No. 1 specimens shall be weighed and measured daily to give data for calculating the moisture content and volume of the specimens each day.

Procedure

5. (*a*) At the end of the storage in the moist room, water-saturated felt pads, blotters or similar absorptive material shall be placed between the specimens and the carriers, and the assembly placed in a refrigerator having a constant temperature not warmer than minus 23 C. (minus 10 F.) for 22 hr. and removed. The No. 1 specimen (volume and moisture change specimen) shall be weighed and measured and both specimens placed in the moist room. Free water shall be made available to the absorbent pads under the specimens to permit absorption of water by the specimens by capillarity.

(*b*) After 22 hr. in the moist room, both specimens shall be weighed and the No. 1 specimen measured. Specimen No. 2 (soil-cement loss specimen) shall then be given two firm strokes on all areas with a wire scratch brush to remove all material loosened during freezing and thawing. The specimen shall be again weighed after brushing. The moisture content of the material brushed from the specimen shall be determined directly, but if this is not possible, it may be assumed to be equal to the moisture content of the No. 1 specimen. The oven-dry (110 C., 230 F.) weight of material brushed from the specimen shall be calculated.

(*c*) The procedures described in Paragraphs (*a*) and (*b*) constitute one cycle (48 hr.) of freezing and thawing. The specimens shall then be replaced in the refrigerator and the procedure continued. The data collected will permit calculations of volume and moisture changes of specimen No. 1 and the soil-cement losses of specimen No. 2.

(*d*) The freezing and thawing of the No. 1 specimens shall be continued for 12 cycles or discontinued prior to 12 cycles should the measurements become inaccurate due to distortion or soil-cement loss of the specimen. The freezing and thawing of the No. 2 specimens shall be continued for 12 cycles, if possible.

Calculation

6. The volume and moisture changes and the soil-cement losses of the specimens shall be calculated as follows:

(*a*) The difference between the volume of specimen No. 1 at time of molding and subsequent volumes shall be calculated as a percentage of the original volume.

(*b*) The moisture content of specimen No. 1 at the time of molding and subsequent moisture contents shall be calculated as a percentage of the original oven-dry weight of the specimen.

(*c*) The soil-cement loss of specimen No. 2 shall be calculated as a percentage of the original oven-dry weight of the specimen.

Standard Specifications for

ASPHALT FOR USE IN CONSTRUCTING BUILT-UP ROOF COVERINGS[1]

A.S.T.M. Designation: D 312 – 44

ADOPTED, 1941; REVISED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 312; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover asphalt intended for use as hot-cement and mopping coat in the construction of built-up roof coverings for roofs surfaced in various manners, laid either over boards or concrete on various inclines.

Primer

2. The material used as a primer when this asphalt is used over concrete and gypsum roof slabs shall be asphalt primer conforming to the Standard Specifications for Primer for Use with Asphalt in Dampproofing and Waterproofing (A.S.T.M. Designation: D 41).[3]

Membrane Materials

3. The felts for use in constructing built-up roof coverings with this asphalt shall be of the type covered by any of the following specifications:

(*a*) Standard Specifications for Asphalt-Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs (A.S.T.M. Designation: D 226),[3]

(*b*) Tentative Specifications for Asphalt-Saturated Asbestos Felts for Use in Waterproofing and in Constructing Built-Up Roofs (A.S.T.M. Designation: D 250),[3] and

(*c*) Tentative Specifications for Asphalt-Saturated and Coated Asbestos Felts for Use in Constructing Built-Up Roofs (A.S.T.M. Designation: D 655).[3]

Types

4. The asphalts covered by these specifications are as follows:

(*a*) Relatively susceptible asphalts with good adhesive and "self-healing" properties for use in slag or gravel surfaced roofing on inclines up to 3 in. per ft.,

(*b*) Somewhat less susceptible asphalts for roofing laid on inclines up to 3 in. per ft. which are not surfaced with slag or gravel, and

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] Prior to adoption as standard, these specifications were published as tentative from 1929 to 1941, being revised in 1935, 1937, and 1939.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(*c*) Relatively nonsusceptible asphalts for use on inclines between 3 and 6 in. per ft.

Properties

5. The asphalt shall be homogeneous and free from water. It shall conform to the requirements prescribed in Table I.

TABLE I.—REQUIREMENTS FOR ASPHALT FOR CONSTRUCTING BUILT-UP ROOF COVERINGS.

	Type of Roofing					
	Mineral Surfaced Flat		Unsurfaced Flat		Unsurfaced Steep	
	Min.	Max.	Min.	Max.	Min.	Max.
For use on decks having inclines:						
Over boards, in. per ft.	....	3	....	3	3	6
Over concrete, in. per ft.:						
With nailing	....	3	....	3	3	6
Without nailing	....	1.5	....	1.5	1.5	3
Softening point (ring-and-ball method)	135 F. (57 C.)	150 F. (66 C.)	135 F. (57 C.)	170 F. (77 C.)	170 F. (77 C.)	200 F. (93 C.)
Flash point (Cleveland open cup)	375 F. (195 C.)	....	375 F. (195 C.)	....	375 F. (195 C.)	....
Penetration:						
0 C. (32 F.), 200 g., 60 sec.	5	....	5	....	10	....
25 C. (77 F.), 100 g., 5 sec.	20	50	20	40	20	40
46 C. (115 F.), 50 g., 5 sec.	100	....	....	150	....	100
Ductility at 25 C. (77 F.), (5 cm. per min.), cm.	10	....	4	....	3	....
Loss on heating at 163 C. (325 F.), 50 g., 5 hr., per cent.	....	1.5	....	1.5	....	1.5
Penetration of residue, per cent of original	60	....	60	....	60	....
Total bitumen soluble in carbon disulfide, per cent:						
Filled or native asphalt	65	....	65	....	65	....
Unfilled asphalt	99	....	99	....	99	....
Proportion of bitumen soluble in carbon tetrachloride, per cent	99	....	99	....	99	....
Ash, per cent:						
Filled or native asphalt	7.5	35	7.5	35	7.5	35
Unfilled asphalt	....	1	....	1	....	
Coarse particles retained on No. 200 (74-micron) sieve as percentage of matter insoluble in carbon disulfide, per cent	....	12	....	12	....	12

Sampling and Methods of Testing

6. The asphalt shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(*a*) *Sampling.*—Tentative Methods of Sampling Bituminous Materials (A.S.T.M. Designation: D 140).[3]

(*b*) *Softening Point.*—Standard Method of Test for Softening Point of Bituminous Materials (Ring-and-Ball Method) (A.S.T.M. Designation: D 36).[3]

(*c*) *Flash Point.*—Standard Method of Test for Flash and Fire Points by Means of Open Cup (A.S.T.M. Designation: D 92).[3]

(*d*) *Penetration.*—Standard Method of Test for Penetration of Bituminous Materials (A.S.T.M. Designation: D 5).[3]

(*e*) *Ductility.*—Standard Method of Test for Ductility of Bituminous Materials (A.S.T.M. Designation: D 113).[3]

(*f*) *Loss on Heating.*—Tentative Method of Test for Loss on Heating of Oil and Asphaltic Compounds (A.S.T.M. Designation: D 6).[3]

(*g*) *Soluble in Carbon Disulfide.*—Standard Method of Test for Determination of Bitumen (A.S.T.M. Designation: D 4).[3]

(*h*) *Bitumen Soluble in Carbon Tetrachloride.*—Standard Method of Test for Proportion of Bitumen Soluble in Carbon Tetrachloride (A.S.T.M. Designation: D 165).[3]

(*i*) *Ash.*—Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (A.S.T.M. Designation: D 271).[4]

(*j*) *Coarse Particles.*—Standard Method of Test for Coarse Particles in Mixtures of Asphalt and Mineral Matter (A.S.T.M. Designation: D 313).[3]

[4] 1944 Book of A.S.T.M. Standards, Part III.

Standard Specifications for

COAL-TAR PITCH FOR ROOFING, DAMPPROOFING, AND WATERPROOFING[1]

A.S.T.M. Designation: D 450 - 41

ADOPTED, 1941.[2]

Reapproved in 1944 Without Change.

This Standard of the American Society for Testing Materials is issued under the fixed designation D 450; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover coal-tar pitch suitable for use as a mopping coat in the construction of built-up roofs surfaced with slag or gravel, as a mopping coat in dampproofing, or as a plying or mopping cement in the construction of a membrane system of waterproofing.

Primer

2. The material used as a primer shall be creosote conforming to the Standard Specifications for Creosote for Priming Coat with Coal-Tar Pitch in Dampproofing and Waterproofing (A.S.T.M. Designation: D 43) of the American Society for Testing Materials.[3]

Membrane Materials

3. (*a*) For the construction of built-up roofs, felts conforming to the Standard Specifications for Coal-Tar Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs (A.S.T.M. Designation: D 227) of the American Society for Testing Materials[3] shall be used.

(*b*) For the construction of a membrane system of waterproofing, any or all of the following felts, fabrics, or burlap conforming to the specifications of the American Society for Testing Materials may be used alone or in various combinations:

Felt.—Standard Specifications for Coal-Tar Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs (A.S.T.M. Designation: D 227).[3]

Cotton Fabrics.—Standard Specifications for Woven Cotton Fabrics Saturated with Bituminous Substances for Use in Waterproofing (A.S.T.M. Designation: D 173),[3] coal-tar saturant.

Types

4. The coal-tar pitches covered by these specifications are of two types, as follows:

Type A.—Coal-tar pitch for use as a

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] Prior to adoption as standard, these specifications were published as tentative from 1937 to 1941, being revised in 1938.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

mopping coat in the construction of built-up roofs surfaced with slag or gravel on inclines not exceeding 3 in. per ft. where nailing is employed, and not exceeding 1 in. per ft. where nailing is omitted.

Coal-tar pitch for use as a mopping coat in dampproofing or as a plying cement in the construction of a membrane system of waterproofing above ground level when not exposed to temperatures exceeding 125 F. (52 C.).

specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(*a*) *Sampling.*—Tentative Methods of Sampling Bituminous Materials (A.S.T.M. Designation: D 140).[3]

(*b*) *Water.*—Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (A.S.T.M. Designation: D 95).[3]

(*c*) *Specific Gravity.*—Standard Method

TABLE I.—REQUIREMENTS FOR COAL-TAR PITCH FOR ROOFING, DAMPPROOFING, AND WATERPROOFING.

	Min.	Max.
Water, per cent	....	0
Specific gravity, 25/25 C. (77/77 F.)	1.22	1.34
Softening point (cube-in-water method):		
Type A	140 F. (60 C.)	155 F. (68 C.)
Type B	120 F. (49 C.)	140 F. (60 C.)
Flash point (Cleveland open cup)	248 F. (120 C.)	....
Distillation test:		
Total distillate by weight 32 to 572 F. (0 to 300 C.), per cent	....	10
Residue by weight, per cent	90	....
Specific gravity, 100/60 F. (38/15.5 C.), of total distillate to 572 F. (300 C.)	1.03	....
Softening point (cube-in-water method) of residue from distillation to 572 F. (300 C.)	....	175 F. (79 C.)
Ductility at 25 C. (77 F.), (5 cm. per min), cm.	50	....
Total bitumen soluble in carbon disulfide, per cent	72	83
Ash, per cent	....	0.5

NOTE.—The usual range for softening point of roofing pitch shall be 140 to 150 F. In hot summer weather a softening point range of 145 to 155 F. will be permitted.

NOTE.—This type of coal-tar pitch is suitable for railroad bridges, tanks, retaining walls, culverts, dams, conduits, etc.

Type B.—Coal-tar pitch for use as a mopping coat in dampproofing or as a plying cement in the construction of a membrane system of waterproofing below ground level under uniformly moderate temperature conditions both during the process of installation and during service.

Properties

5. The coal-tar pitch shall be homogenous and shall conform to the requirements prescribed in Table I.

Methods of Sampling and Testing

6. The coal-tar pitch shall be sampled and the properties enumerated in these

of Test for Specific Gravity of Road Oils, Road Tars, Asphalt Cements, and Soft Tar Pitches (A.S.T.M. Designation: D 70).[3]

(*d*) *Softening Point.*—Standard Method of Test for Softening Point of Tar Products (Cube-in-Water Method) (A.S.T.M. Designation: D 61).[2]

(*e*) *Flash Point.*—Standard Method of Test for Flash and Fire Points by Means of Open Cup (A.S.T.M. Designation: D 92).[3]

(*f*) *Distillation.*—Standard Method of Test for Distillation of Tar Products Suitable for Road Treatment (A.S.T.M. Designation: D 20).[3]

(*g*) *Ductility.*—Standard Method of Test for Ductility of Bituminous Materials (A.S.T.M. Designation: D 113).[3]

(*h*) *Bitumen Soluble in Carbon Disulfide.*—Standard Method of Test for Determination of Bitumen (A.S.T.M. Designation: D 4).[3]

(*i*) *Ash.*—Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (A.S.T.M. Designation: D 271).[4]

[4] 1944 Book of A.S.T.M. Standards, Part III.

AMERICAN ASSOCIATION STATE
HIGHWAY OFFICIALS STANDARD
A.A.S.H.O. NO.: M 116-42

Standard Specifications for

PRIMER FOR USE WITH ASPHALT IN DAMPPROOFING AND WATERPROOFING[1]

A.S.T.M. Designation: D 41 - 41

ADOPTED, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 41; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover asphaltic primer for use, when specified, with asphalt in dampproofing and waterproofing below or above ground level, for application to concrete and masonry surfaces.

Properties

2. (*a*) The primer shall be free from water and shall conform to the following requirements:

Furol viscosity at 25 C. (77 F.)...25 to 150 sec.
Distillation, per cent by volume:
Up to 225 C. (437 F.).....not less than 35
Up to 360 C. (680 F.).....not more than 65

(*b*) The residue obtained from the distillation up to 360 C. (680 F.) shall conform to the following requirements:

Penetration at 25 C. (77 F.), 100 g., 5 sec.....20 to 50
Matter soluble in carbon disulfide....not less than 99 per cent

Sampling

3. (*a*) From each shipment or fraction thereof representing primer of the same kind, a number of packages shall be selected at random equivalent to the cube root of the total number of packages in the lot. If the cube root as calculated proves to be a fractional number, it shall be expressed as the next higher whole number. For convenience, the following table is given, showing the number of samples to be selected for shipments of various sizes:

Packages in Shipment	Packages Selected
2 to 8	2
9 to 27	3
28 to 64	4
65 to 125	5
126 to 216	6
217 to 343	7
344 to 512	8
513 to 729	9
730 to 1000	10
1001 to 1331	11

(*b*) By means of a paddle, the contents of each package selected shall be thor-

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] Prior to their present adoption as standard, these specifications were published as tentative from 1917 to 1925, being revised in 1922 and 1925. They were adopted in 1925, published as standard from 1925 to 1936, being revised in 1926, but were withdrawn and republished as tentative from 1936 to 1941, being revised in 1939.

oughly stirred so as to bring all portions thereof into uniform distribution. With an appropriate sampler, not less than 1 pt. of the primer shall be immediately drawn from the center of each package, and transferred to a clean receptacle of suitable size, which in turn shall be kept tightly covered, prior and immediately after the samples are being introduced. After all the pint samples have been added, the contents of the container shall be thoroughly agitated, whereupon 1 qt. of the mixture shall immediately be transferred to a clean quart can provided with a tightly fitting screw cap, which shall be immediately fastened in place. This sample shall be considered as representative of the shipment and shall be transmitted to the laboratory for examination, bearing a suitable tag or other means of identification.

Methods of Testing

4. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials:

(*a*) *Furol Viscosity.* — Standard Method of Test for Viscosity by Means of the Saybolt Viscosimeter (A.S.T.M. Designation: D 88).[3]

(*b*) *Distillation.*—Standard Method of Test for Distillation of Cut-Back Asphaltic Products (A.S.T.M. Designation: D 402).[3]

(*c*) *Penetration.*—Standard Method of Test for Penetration of Bituminous Materials (A.S.T.M. Designation: D 5).[3]

(*d*) *Bitumen Soluble in Carbon Disulfide.*—Standard Method of Test for Determination of Bitumen (A.S.T.M. Designation: D 4).[3]

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[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for

CREOSOTE FOR PRIMING COAT WITH COAL-TAR PITCH IN DAMPPROOFING AND WATERPROOFING[1]

A.S.T.M. Designation: D 43 – 41

ADOPTED, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 43; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover creosote primer for use, when specified, with coal-tar pitch in dampproofing and waterproofing below or above ground level, for application to concrete and masonry surfaces.

Properties[2a]

2. Creosote primer shall be a distillate of coal-gas tar or coke-oven tar and shall conform to the following requirements:

Water	not more than 1 per cent
Consistency at 5 C. (41 F.)	entirely fluid and crystal free[a]
Specific gravity, 38/15.5 C. (100/60 F.)	not less than 1.06
Matter insoluble in benzol	not more than 0.5 per cent
Distillation, based on water-free oil:	
Up to 210 C.	not more than 1 per cent
Up to 235 C.	not more than 10 per cent
Up to 355 C.	not less than 65 per cent
Coke residue	not more than 2 per cent

[a] The creosote shall be rated as crystal free if no crystals are formed when 100 ml. of the sample is maintained at a temperature of 5 C. (41 F.) for 3 hr., in a 125 ml. Erlenmeyer flask, with occasional stirring.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] Prior to their present adoption as standard, these specifications were published as tentative from 1917 to 1925, being revised in 1922. They were adopted in 1925, published as standard from 1925 to 1936, but were withdrawn and republished as tentative from 1936 to 1941, being revised in 1937.

[2a] Editorially revised in 1942.

Sampling

3. (*a*) From each shipment or fraction thereof representing creosote of the same kind, a number of packages shall be selected at random equivalent to the cube root of the total number of packages in the lot. If the cube root as calculated proves to be a fractional number, it shall be expressed as the next higher whole number. For convenience, the following table is given, showing the number of samples to be selected for shipments of various sizes:

Packages in Shipment	Number of Packages Selected
2 to 8	2
9 to 27	3
28 to 64	4
65 to 125	5
126 to 216	6
217 to 343	7
344 to 512	8
513 to 729	9
730 to 1000	10
1001 to 1331	11

(*b*) By means of a paddle, the contents of each package selected shall be thoroughly stirred so as to bring all portions thereof into uniform distribution. With

an appropriate sampler, not less than 1 pt. of the creosote shall be immediately drawn from the center of each package and transferred to a clean receptacle of suitable size, which in turn shall be kept tightly covered, prior and immediately after the samples are being introduced. After all the pint samples have been added, the contents of the container shall be thoroughly agitated, whereupon 1 qt. of the mixture shall immediately be transferred to a clean quart can provided with a tightly fitting screw cap, which shall be immediately fastened in place. This sample shall be considered as representative of the shipment and shall be transmitted to the laboratory for examination, bearing a suitable tag or other means of identification.

(*c*) Creosote primer in large shipments shall be sampled in accordance with the Standard Methods of Sampling and Testing Creosote (A.S.T.M. Designation: D 38) of the American Society for Testing Materials.[3]

Methods of Testing

4. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials:

(*a*) *Water.*—Standard Method of Test for Water in Creosote (A.S.T.M. Designation: D 370).[3]

(*b*) *Specific Gravity.* — Standard Method of Test for Specific Gravity of Creosote (A.S.T.M. Designation D 368).[3]

(*c*) *Matter Insoluble in Benzol.*—Standard Methods of Test for Insoluble Matter in Creosote (A.S.T.M. Designation: D 367).[3]

(*d*) *Distillation.*—Standard Method of Test for Distillation of Creosote (A.S.T.M. Designation: D 246).[3]

(*e*) *Coke Residue.*—Standard Method of Test for Coke Residue of Creosote (A.S.T.M. Designation: D 168).[3]

(*f*) *Volume and Specific Gravity Correction.*—Standard Volume and Specific Gravity Correction Tables for Creosote and Coal Tar (A.S.T.M. Designation: D 347).[3]

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Method of Test for

STEAM DISTILLATION OF BITUMINOUS PROTECTIVE COATINGS[1]

A.S.T.M. Designation: D 255 - 28

ADOPTED, 1928.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 255; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This is a general method for the separation and recovery of the solvent and the base in bituminous mixtures. The bituminous mixture is distilled in a current of steam, the solvent condensed and separated from the water.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Steam Generator.*—A steam generator made of either metal or glass, with a capacity of from 2 to 4 liters, suitable for continued use in the production of steam. If of glass, it shall be fitted with two outlets with suitable connections for rubber tubing. In the case of a metal generator, a large opening for filling and a water gage shall be additional parts of the apparatus. The generator shall be supplied with suitable pinch cocks or valves so that steam may be blown off to the atmosphere until the test is ready.

(*b*) *Bath.*—A bath made of metal and having sufficient capacity to permit immersion of the distilling flask to a depth of not less than 10 cm.

(*c*) *Source of Heat.*—A gas burner, electric hot plate, or equivalent source of heat for the bath.

(*d*) *Distilling Flask.*—A 100-ml. distilling flask with a short ring neck and round-bottom. It shall be fitted with a three-hole rubber stopper, a steam delivery tube which will reach to within $\frac{1}{2}$ in. (12.7 mm.) of the bottom of the flask and project from the top to a convenient distance for connection to the generator, a vapor outlet tube which extends from beneath the rubber stopper to a point sufficiently above the distilling flask to permit convenient connection to the condenser, and a thermometer. The steam tubing should be not less than 2 nor more than 4 mm. in internal diameter and the vapor outlet tube should be not less than 5 mm. in internal diameter.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] Prior to adoption as standard, this method was published as tentative from 1926 to 1928. Editorially revised and rearranged in 1939.

(*e*) *Condenser.*—A condenser made of No. 20 Stubbs gage seamless brass tubing $\frac{9}{16}$ in. (14.29 mm.) in outside diameter and 22 in. (55.88 cm.) in length. It shall be set at an angle of 75 deg. from the perpendicular and be surrounded with a cooling bath 15 in. (38.1 cm.) in length, approximately 4 in. (10.16 cm.) in width by 6 in. (15.24 cm.) in height. The lower end of the condenser tube shall be cut off at an acute angle, and curved downward for a length of 3 in. (7.62 cm.) and slightly backward so as to insure contact with the wall of the graduate at a point 1 to $1\frac{1}{4}$ in. (2.54 to 3.18 cm.) below the top of the graduate when it is in position to receive the distillate.

(*f*) *Separatory Funnel.*—A separatory funnel as shown in Fig. 1, having a capacity of not less than 500 ml.

(*g*) *Thermometer.*—An A.S.T.M. Low Distillation Thermometer, total immersion, graduated in either Centigrade or Fahrenheit degrees as specified, having a range of 0 to 300 C. or 30 to 580 F. and conforming to the requirements for this thermometer as prescribed in the Standard Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1) of the American Society for Testing Materials.[3]

(*h*) *Accessories.*—Accessories consisting of suitable ring stands for supporting the steam generator, distilling flask, bath for distilling flask, and separatory funnel.

Procedure

3. The apparatus shall be assembled as shown in Fig. 1. The steam generator shall be filled with water and heat applied. The bath shall be filled with a high-flash-point oil and raised to approximately 140 C. (284 F.). A 500-ml. sample shall be weighed into the round-bottom flask. The steam generator shall be connected to the steam delivery tube, the end of which shall be within $\frac{1}{2}$ in. of the bottom of the distilling flask. The outlet from the distilling flask shall be connected to the condenser and the separatory funnel placed in position at the outlet of the condenser to receive the distillate. The end of the bulb of the thermometer in the steam distilling flask shall be placed within $\frac{1}{2}$ in. (12.7 mm.) of the bottom of the distilling flask. When the temperature of the

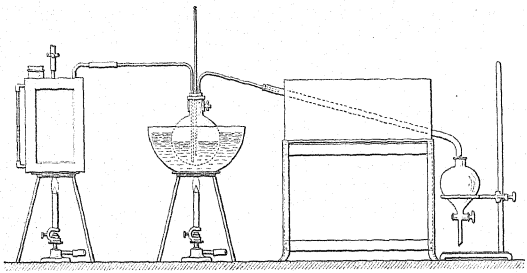


FIG. 1.—Assembly of Apparatus.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

sample in the distilling flask reaches 130 C. the outlet of the steam generator shall be closed, thus forcing the steam to pass through the sample. The flow of steam shall be adjusted so that the distillate is collected at the rate of approximately 6 to 10 ml. per min. The distillation shall be stopped when 100 ml. of the distillate contains not more than 0.5 ml. of solvent as determined by measuring the amount of oil in 100 ml. of distillate. When the distillation is finished, the water shall be separated from the distillate and the distillate measured and retained for further tests if required by the specifications. In some cases, the distillate does not separate readily from the water; this separation can be facilitated by the addition of sodium chloride which will result in a sufficient difference in gravity to produce a clear separation of the two layers.

Report

4. The results shall be reported as a percentage by weight or by volume as required by the specifications, based on the weight of the sample taken.

Standard Methods of

TESTING FILMS DEPOSITED FROM BITUMINOUS EMULSIONS[1]

A.S.T.M. Designation: D 466 – 42

ADOPTED, 1942.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 466; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

RESISTANCE TO FLOW UNDER HEAT (SLIDE TEST)

Scope

1. The slide test is applicable to bituminous emulsions or reinforced emulsions intended to be applied at the rate of not less than 3 gal. per 100 sq. ft. and on substantially vertical surfaces.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Brass Mask.*—A brass mask approximately $\frac{7}{64}$ in. in thickness with a rectangular opening 4 by 4 in.

(*b*) *Tiles.*—Unglazed ceramic tiles[3] white, nonvitreous, dust pressed body with an absorption range of 15 to 18 per cent (determined in accordance with the Standard Methods of Sampling and Testing Structural Clay Tile (A.S.T.M. Designation: C 112) of the American Society for Testing Materials[4]) approximately 6 by 6 in. by $\frac{3}{8}$ to $\frac{1}{2}$ in. in thickness, or of sufficient size to accommodate the opening of the mask.

Procedure

3. (*a*) *Preparation of Tile.*—The tile shall be immersed in distilled water for at least 10 min. and the excess water removed immediately before application of the emulsion.

(*b*) *Preparation of Film.*—The sample of emulsion shall be thoroughly stirred. The brass mask shall then be applied to the smooth face of the tile and the emulsion spread over the area of the tile within the mask opening. The excess emulsion shall be doctored off with a flat scraper so that the film after drying shall be not less than 0.04 in. (1 mm.) in thickness.

(*c*) *Drying.*—The coated tile shall then be dried to constant weight in an atmosphere of low relative humidity at room temperature and weighed every 24 hr. A film shall be considered dry when the loss in two successive 24-hr. weighings is not greater than 0.1 g.

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] Prior to adoption as standard, these methods were published as tentative from 1937 to 1942, being revised in 1938 and 1942.

[3] Suitable unglazed tile can be obtained from the Robertson Art Tile Co., Morrisville, Pa., and from Gladding-McBean and Co., Los Angeles, Calif.

[4] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

NOTE.—Drying may be hastened by placing the specimen in a current of air from an electric fan.

(*d*) *Testing.*—After drying and removing the brass mask, the coated tile shall be suspended vertically in the center of an air oven maintained at 176 ± 5 F. (80 ± 3 C.). The internal dimensions of the oven shall be not less than 12 by 12 by 12 in. An electrically heated oven is recommended. A thermometer shall be inserted in the oven to such depth that its bulb will be in line with the center of the specimen. The tile shall be maintained at the prescribed temperature for exactly 2 hr. Any sliding shall be determined by a reference line placed originally on the tile, coincident with the lower edge of the dried film.

RESISTANCE TO WATER ACTION

Scope

4. The purpose of this test is to determine the ability of the dried film to retain its adhesion and to resist re-emulsification after immersion in water.

Procedure

5. (*a*) *Preparation of Film.*—The film to be tested shall be prepared as described in Sections 2 and 3.

(*b*) *Testing.*—After drying, a ring approximately 2 in. in diameter and 1 in. in height shall be pressed into the surface of the coated tile. The ring shall be filled with tap water and the test specimen maintained at room temperature between 70 to 90 F. (21 to 32 C.). After 24 hr. the character of the bituminous film shall be noted by cutting and attempting to lift a portion of the submerged film with a knife or teasing needle. Adhesion or bond shall be determined by making intersecting cuts with the knife or needle and lifting the cut film at the point of intersection. Re-emulsification is indicated if the water becomes darkened by rubbing the submerged surface of the uncut film lightly with a rubber policeman.

STANDARD VOLUME CORRECTION TABLE FOR TAR AND COAL-TAR PITCH[1]

A.S.T.M. Designation: D 633 - 44

ADOPTED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 633; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

This table has been prepared by the National Bureau of Standards to meet a demand from the tar industry for a short and convenient table for reducing volumes of road tar and coal-tar pitch to the basis of 60 F., when extreme accuracy is not required. The table shows the volume occupied at 60 F. by a quantity of material occupying unit volume at the indicated temperature.

The groups and coefficients of expansion for the several subdivisions of the present table are as follows:

Group Number	Coefficient of Expansion, per Deg. Fahr. at 60 F.	Material
000	0.00026	Coal-Tar Pitch for Roofing, Dampproofing, and Waterproofing[a] Coal-Tar Pitch for Stone Block Filler[b]
00	0.00030	Tar,[c] Grades RT-5, RT-6, RT-7, RT-8, RT-9, RT-10, RT-11, RT-12, RT.C.B.-5, and RT.C.B.-6
0	0.00035	Tar,[c] Grades RT-1, RT-2, RT-3, and RT-4

[a] Standard Specifications for Coal-Tar Pitch for Roofing, Dampproofing, and Waterproofing (A.S.T.M. Designation: D 450) of the American Society for Testing Materials.[3]

[b] Standard Specifications for Coal-Tar Pitch for Stone Block Filler (A.S.T.M. Designation: D 112) of the American Society for Testing Materials.[3]

[c] Tentative Specifications for Tar (A.S.T.M. Designation: D 490) of the American Society for Testing Materials.[3]

NOTE: *Example.*—The values given in the table are in the form of multipliers; that is, the volume at the indicated temperature for each group, multiplied by the corresponding factor in the table, equals the volume at 60 F. For example, if the volume at 238 F. of coal-tar roofing pitch (Group 000) equals 7900 gal., then the volume at 60 F. equals 7900 × 0.9558 or 7551 gal.

[1] Under the standardization procedure of the Society, this standard is under the joint jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials and Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] Prior to adoption as standard, this table was published as tentative from 1941 to 1944.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

VOLUME CORRECTION TABLE FOR TAR AND COAL-TAR PITCH.

Group 000

Legend: t = observed temperature in degrees Fahrenheit; M = multiplier for reducing volumes to the basis of 60 F.

t	M	t	M	t	M	t	M	t	M	t	M	t	M
0	1.0158	35	1.0065	70	0.9974	105	0.9884	140	0.9796	175	0.9710	210	0.9625	245	0.9541
1	1.0156	36	1.0063	71	0.9971	106	0.9882	141	0.9794	176	0.9707	211	0.9622	246	0.9539
2	1.0153	37	1.0060	72	0.9969	107	0.9879	142	0.9791	177	0.9705	212	0.9620	247	0.9536
3	1.0150	38	1.0058	73	0.9966	108	0.9877	143	0.9789	178	0.9702	213	0.9617	248	0.9534
4	1.0148	39	1.0055	74	0.9964	109	0.9874	144	0.9786	179	0.9700	214	0.9615	249	0.9532
5	1.0145	40	1.0052	75	0.9961	110	0.9872	145	0.9784	180	0.9697	215	0.9613		
6	1.0142	41	1.0050	76	0.9959	111	0.9869	146	0.9781	181	0.9695	216	0.9610		
7	1.0140	42	1.0047	77	0.9956	112	0.9867	147	0.9779	182	0.9693	217	0.9608		
8	1.0137	43	1.0044	78	0.9953	113	0.9864	148	0.9776	183	0.9690	218	0.9605		
9	1.0134	44	1.0042	79	0.9951	114	0.9862	149	0.9774	184	0.9688	219	0.9603		
10	1.0132	45	1.0039	80	0.9948	115	0.9859	150	0.9771	185	0.9685	220	0.9601		
11	1.0129	46	1.0036	81	0.9946	116	0.9856	151	0.9769	186	0.9683	221	0.9598		
12	1.0126	47	1.0034	82	0.9943	117	0.9854	152	0.9766	187	0.9680	222	0.9596		
13	1.0124	48	1.0031	83	0.9941	118	0.9851	153	0.9764	188	0.9678	223	0.9593		
14	1.0121	49	1.0029	84	0.9938	119	0.9849	154	0.9761	189	0.9675	224	0.9591		
15	1.0118	50	1.0026	85	0.9935	120	0.9846	155	0.9759	190	0.9673	225	0.9589		
16	1.0116	51	1.0024	86	0.9933	121	0.9844	156	0.9756	191	0.9671	226	0.9586		
17	1.0113	52	1.0021	87	0.9930	122	0.9841	157	0.9754	192	0.9668	227	0.9584		
18	1.0110	53	1.0018	88	0.9928	123	0.9839	158	0.9752	193	0.9666	228	0.9581		
19	1.0108	54	1.0016	89	0.9925	124	0.9836	159	0.9749	194	0.9663	229	0.9579		
20	1.0105	55	1.0013	90	0.9923	125	0.9834	160	0.9747	195	0.9661	230	0.9577		
21	1.0102	56	1.0010	91	0.9920	126	0.9831	161	0.9744	196	0.9658	231	0.9574		
22	1.0100	57	1.0008	92	0.9917	127	0.9829	162	0.9742	197	0.9656	232	0.9572		
23	1.0097	58	1.0005	93	0.9915	128	0.9826	163	0.9739	198	0.9654	233	0.9570		
24	1.0094	59	1.0003	94	0.9912	129	0.9824	164	0.9737	199	0.9651	234	0.9567		
25	1.0092	60	1.0000	95	0.9910	130	0.9821	165	0.9734	200	0.9649	235	0.9565		
26	1.0089	61	0.9997	96	0.9907	131	0.9819	166	0.9732	201	0.9646	236	0.9562		
27	1.0087	62	0.9995	97	0.9905	132	0.9816	167	0.9729	202	0.9644	237	0.9560		
28	1.0084	63	0.9992	98	0.9902	133	0.9814	168	0.9727	203	0.9642	238	0.9558		
29	1.0081	64	0.9990	99	0.9900	134	0.9811	169	0.9724	204	0.9639	239	0.9555		
30	1.0079	65	0.9987	100	0.9897	135	0.9809	170	0.9722	205	0.9637	240	0.9553		
31	1.0076	66	0.9984	101	0.9895	136	0.9806	171	0.9719	206	0.9634	241	0.9551		
32	1.0073	67	0.9982	102	0.9892	137	0.9804	172	0.9717	207	0.9632	242	0.9548		
33	1.0071	68	0.9979	103	0.9889	138	0.9801	173	0.9715	208	0.9629	243	0.9546		
34	1.0068	69	0.9977	104	0.9887	139	0.9799	174	0.9712	209	0.9627	244	0.9543		

VOLUME CORRECTION TABLE FOR TAR AND COAL-TAR PITCH.

Group 00

Legend: t = observed temperature in degrees Fahrenheit; M = multiplier for reducing volumes to the basis of 60 F.

t	M	t	M	t	M	t	M	t	M	t	M	t	M	t	M
0	1.0183	35	1.0076	70	0.9970	105	0.9867	140	0.9766	175	0.9667	210	0.9569	245	0.9474
1	1.0180	36	1.0073	71	0.9967	106	0.9864	141	0.9763	176	0.9664	211	0.9567	246	0.9471
2	1.0177	37	1.0069	72	0.9964	107	0.9861	142	0.9760	177	0.9661	212	0.9564	247	0.9469
3	1.0174	38	1.0066	73	0.9961	108	0.9858	143	0.9757	178	0.9658	213	0.9561	248	0.9466
4	1.0171	39	1.0063	74	0.9958	109	0.9855	144	0.9754	179	0.9655	214	0.9558	249	0.9463
5	1.0168	40	1.0060	75	0.9955	110	0.9852	145	0.9751	180	0.9653	215	0.9556	.....	.....
6	1.0165	41	1.0057	76	0.9952	111	0.9849	146	0.9748	181	0.9650	216	0.9553	.....	.....
7	1.0162	42	1.0054	77	0.9949	112	0.9846	147	0.9746	182	0.9647	217	0.9550	.....	.....
8	1.0158	43	1.0051	78	0.9946	113	0.9843	148	0.9743	183	0.9644	218	0.9547	.....	.....
9	1.0155	44	1.0048	79	0.9943	114	0.9841	149	0.9740	184	0.9641	219	0.9545	.....	.....
10	1.0152	45	1.0045	80	0.9940	115	0.9838	150	0.9737	185	0.9639	220	0.9542	.....	.....
11	1.0149	46	1.0042	81	0.9937	116	0.9835	151	0.9734	186	0.9636	221	0.9539	.....	.....
12	1.0146	47	1.0039	82	0.9934	117	0.9832	152	0.9731	187	0.9633	222	0.9537	.....	.....
13	1.0143	48	1.0036	83	0.9931	118	0.9829	153	0.9729	188	0.9630	223	0.9534	.....	.....
14	1.0140	49	1.0033	84	0.9929	119	0.9826	154	0.9726	189	0.9627	224	0.9531	.....	.....
15	1.0137	50	1.0030	85	0.9926	120	0.9823	155	0.9723	190	0.9625	225	0.9528	.....	.....
16	1.0134	51	1.0027	86	0.9923	121	0.9820	156	0.9720	191	0.9622	226	0.9526	.....	.....
17	1.0131	52	1.0024	87	0.9920	122	0.9817	157	0.9717	192	0.9619	227	0.9523	.....	.....
18	1.0128	53	1.0021	88	0.9917	123	0.9815	158	0.9714	193	0.9616	228	0.9520	.....	.....
19	1.0125	54	1.0018	89	0.9914	124	0.9812	159	0.9712	194	0.9614	229	0.9517	.....	.....
20	1.0121	55	1.0015	90	0.9911	125	0.9809	160	0.9709	195	0.9611	230	0.9515	.....	.....
21	1.0118	56	1.0012	91	0.9908	126	0.9806	161	0.9706	196	0.9608	231	0.9512	.....	.....
22	1.0115	57	1.0009	92	0.9905	127	0.9803	162	0.9703	197	0.9605	232	0.9509	.....	.....
23	1.0112	58	1.0006	93	0.9902	128	0.9800	163	0.9700	198	0.9602	233	0.9507	.....	.....
24	1.0109	59	1.0003	94	0.9899	129	0.9797	164	0.9697	199	0.9600	234	0.9504	.....	.....
25	1.0106	60	1.0000	95	0.9896	130	0.9794	165	0.9695	200	0.9597	235	0.9501	.....	.....
26	1.0103	61	0.9997	96	0.9893	131	0.9791	166	0.9692	201	0.9594	236	0.9498	.....	.....
27	1.0100	62	0.9994	97	0.9890	132	0.9789	167	0.9689	202	0.9591	237	0.9496	.....	.....
28	1.0097	63	0.9991	98	0.9887	133	0.9786	168	0.9686	203	0.9589	238	0.9493	.....	.....
29	1.0094	64	0.9988	99	0.9884	134	0.9783	169	0.9683	204	0.9586	239	0.9490	.....	.....
30	1.0091	65	0.9985	100	0.9881	135	0.9780	170	0.9681	205	0.9583	240	0.9488	.....	.....
31	1.0088	66	0.9982	101	0.9878	136	0.9777	171	0.9678	206	0.9580	241	0.9485	.....	.....
32	1.0085	67	0.9979	102	0.9876	137	0.9774	172	0.9675	207	0.9578	242	0.9482	.....	.....
33	1.0082	68	0.9976	103	0.9873	138	0.9771	173	0.9672	208	0.9575	243	0.9480	.....	.....
34	1.0079	69	0.9973	104	0.9870	139	0.9768	174	0.9669	209	0.9572	244	0.9477	.....	.....

VOLUME CORRECTION TABLE FOR TAR AND COAL-TAR PITCH.

Group 0

Legend: t = observed temperature in degrees Fahrenheit; M = multiplier for reducing volumes to the basis of 60 F.

t	M	t	M	t	M	t	M	t	M	t	M	t	M
0....	1.0211	35...	1.0088	70..	0.9965	105..	0.9844	140..	0.9724	175..	0.9606	210..	0.9490	245..	0.9376
1....	1.0208	36...	1.0084	71..	0.9962	106..	0.9841	141..	0.9721	176..	0.9603	211..	0.9487	246..	0.9373
2....	1.0204	37...	1.0081	72..	0.9958	107..	0.9837	142..	0.9718	177..	0.9600	212..	0.9484	247..	0.9370
3....	1.0201	38...	1.0077	73..	0.9955	108..	0.9834	143..	0.9714	178..	0.9596	213..	0.9481	248..	0.9367
4....	1.0197	39...	1.0074	74..	0.9952	109..	0.9831	144..	0.9711	179..	0.9593	214..	0.9477	249..	0.9364
5....	1.0194	40...	1.0070	75..	0.9948	110..	0.9827	145..	0.9707	180..	0.9590	215..	0.9474	.....	.....
6....	1.0190	41...	1.0067	76..	0.9944	111..	0.9823	146..	0.9704	181..	0.9586	216..	0.9471	.....	.....
7....	1.0186	42...	1.0063	77..	0.9941	112..	0.9820	147..	0.9701	182..	0.9583	217..	0.9468	.....	.....
8....	1.0183	43...	1.0059	78..	0.9938	113..	0.9816	148..	0.9697	183..	0.9580	218..	0.9464	.....	.....
9....	1.0179	44...	1.0056	79..	0.9934	114..	0.9813	149..	0.9694	184..	0.9576	219..	0.9461	.....	.....
10....	1.0176	45...	1.0052	80..	0.9931	115..	0.9809	150..	0.9691	185..	0.9573	220..	0.9458	.....	.....
11....	1.0172	46...	1.0049	81..	0.9927	116..	0.9806	151..	0.9687	186..	0.9569	221..	0.9454	.....	.....
12....	1.0168	47...	1.0045	82..	0.9924	117..	0.9802	152..	0.9684	187..	0.9566	222..	0.9451	.....	.....
13....	1.0165	48...	1.0042	83..	0.9920	118..	0.9799	153..	0.9680	188..	0.9563	223..	0.9448	.....	.....
14....	1.0161	49...	1.0039	84..	0.9917	119..	0.9795	154..	0.9677	189..	0.9559	224..	0.9445	.....	.....
15....	1.0158	50...	1.0035	85..	0.9914	120..	0.9792	155..	0.9674	190..	0.9556	225..	0.9441	.....	.....
16....	1.0154	51...	1.0032	86..	0.9910	121..	0.9789	156..	0.9670	191..	0.9553	226..	0.9438	.....	.....
17....	1.0151	52...	1.0028	87..	0.9907	122..	0.9785	157..	0.9667	192..	0.9549	227..	0.9435	.....	.....
18....	1.0147	53...	1.0025	88..	0.9903	123..	0.9782	158..	0.9664	193..	0.9546	228..	0.9432	.....	.....
19....	1.0144	54...	1.0021	89..	0.9900	124..	0.9779	159..	0.9660	194..	0.9543	229..	0.9428	.....	.....
20....	1.0141	55...	1.0017	90..	0.9896	125..	0.9775	160..	0.9657	195..	0.9539	230..	0.9425	.....	.....
21....	1.0137	56...	1.0014	91..	0.9892	126..	0.9772	161..	0.9654	196..	0.9536	231..	0.9422	.....	.....
22....	1.0134	57...	1.0010	92..	0.9889	127..	0.9768	162..	0.9650	197..	0.9533	232..	0.9419	.....	.....
23....	1.0130	58...	1.0007	93..	0.9886	128..	0.9765	163..	0.9647	198..	0.9530	233..	0.9415	.....	.....
24....	1.0126	59...	1.0003	94..	0.9882	129..	0.9762	164..	0.9643	199..	0.9527	234..	0.9412	.....	.....
25....	1.0123	60...	1.0000	95..	0.9879	130..	0.9758	165..	0.9640	200..	0.9523	235..	0.9409	.....	.....
26....	1.0119	61...	0.9997	96..	0.9876	131..	0.9755	166..	0.9637	201..	0.9520	236..	0.9406	.....	.....
27....	1.0116	62...	0.9993	97..	0.9872	132..	0.9751	167..	0.9633	202..	0.9517	237..	0.9402	.....	.....
28....	1.0112	63...	0.9990	98..	0.9869	133..	0.9748	168..	0.9630	203..	0.9513	238..	0.9399	.....	.....
29....	1.0109	64...	0.9986	99..	0.9865	134..	0.9745	169..	0.9627	204..	0.9510	239..	0.9396	.....	.....
30....	1.0106	65...	0.9982	100..	0.9862	135..	0.9741	170..	0.9623	205..	0.9507	240..	0.9392	.....	.....
31....	1.0102	66...	0.9979	101..	0.9858	136..	0.9738	171..	0.9620	206..	0.9504	241..	0.9389	.....	.....
32....	1.0098	67...	0.9976	102..	0.9855	137..	0.9735	172..	0.9616	207..	0.9500	242..	0.9386	.....	.....
33....	1.0095	68...	0.9972	103..	0.9852	138..	0.9731	173..	0.9613	208..	0.9497	243..	0.9383	.....	.....
34....	1.0092	69...	0.9969	104..	0.9848	139..	0.9728	174..	0.9610	209..	0.9494	244..	0.9380	.....	.....

Standard Specifications for

ASPHALT MASTIC FOR USE IN WATERPROOFING[1]

ASPHALT CEMENT, MINERAL FILLER, MINERAL AGGREGATE

A.S.T.M. Designation: D 491 - 41

ADOPTED, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 491; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. (*a*) These specifications cover materials for asphalt mastic suitable for use in waterproofing, consisting of asphalt cement, mineral filler, and mineral aggregate.

(*b*) The asphalt mastic covered by these specifications is suitable for waterproofing the floors of buildings and bridges, for reservoirs, waterways, subways, and similar structures. It is not intended for use as a pavement for either foot or vehicular traffic.

Character of Mastic

2. (*a*) The asphalt mastic shall consist of a mixture of asphalt cement, mineral filler, and mineral aggregate which, at a temperature of approximately 400 F. (205 C.), shall be sufficiently plastic to be poured into place and compressed with a wooden trowel or by other suitable means into a compact, watertight sheet of the thickness desired.

(*b*) The raw materials for the asphalt mastic are expected to be mixed on the site of the work, in either suitable mechanically operated heaters and mixers, or by manual stirring in open pans or kettles; or the asphalt cement and mineral matter may be mixed elsewhere and brought to the site of the work in the form of cakes (weighing approximately 75 lb. each) and there combined by the method described for raw materials with mineral aggregate and asphalt cement as may be necessary to comply with the requirements of Section 4. The mixture so prepared shall be applied to the work, or preformed for application to the work, while it is sufficiently hot to be manipulated with hand tools or by other equivalent means.

Properties of Asphalt Cement

3. The asphalt cement used in the asphalt mastic shall conform to the following requirements:

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] Prior to adoption as standard, these specifications were published as tentative from 1938 to 1941, being revised in 1939.

These specifications comprise a consolidation of and replace the former Standard Specifications for Asphalt Mastic for Use in Waterproofing (D 169 - 25), Acid-Resisting Asphalt Mastic (D 223 - 30), and Tentative Specifications for Asphalt Mastic for Use in Waterproofing (Asphalt Cement, Mineral Filler, Mineral Aggregate) (D 491 - 38 T), which standards were accordingly discontinued in 1939.

	Minimum	Maximum
Softening point (ring-and-ball method)	130 F. (54 C.)	150 F. (65 C.)
Penetration at 77 F. (25 C.), 100 g., 5 sec.	20	40
Ductility at 77 F. (25 C.) (5 cm. per min.), cm.	15	..
Loss on heating at 325 F. (163 C., 50 g., 5 hr., per cent	..	2
Penetration of residue at 77 F. (25 C.), per cent of original	60	..
Solubility in carbon disulfide:		
Filled or native asphalt cement, per cent	60	..
Unfilled asphalt cement, per cent	99	..
Bitumen soluble in carbon tetrachloride, per cent	99	..
Ash:		
Filled or native asphalt cement, per cent	7.5	35
Unfilled asphalt cement, per cent	..	1

Properties of Asphalt Mastic

4. (*a*) The asphalt mastic shall conform to the following requirements by weight (or as prescribed by the purchaser within these limitations) as determined by laboratory analysis:

	Minimum	Maximum
Bituminous matter soluble in pure benzol, per cent	10	12
Mineral filler insoluble in benzol, passing a No. 200 (74-micron) sieve, per cent	10	25
Mineral aggregate:		
Passing a No. 40 (420-micron) and retained on a No. 200 (74-micron) sieve, per cent	10	30
Passing a No. 10 (2000-micron) and retained on a No. 40 (420-micron) sieve, per cent	10	35
Passing a No. 4 (4760-micron) and retained on a No. 10 (2000-micron) sieve, per cent	5	25
Passing a ½-in. and retained on a No. 4 (4760-micron) sieve, per cent	5	25

(*b*) The portion of asphalt mastic which is soluble in pure benzol, after extraction and recovery, shall conform to the following requirements:

	Minimum	Maximum
Softening point (ring-and-ball method)	122 F. (50 C.)	158 F. (70 C.)
Penetration at 77 F. (25 C.), 100 g., 5 sec.	15	40
Ductility at 77 F. (25 C.) (5 cm. per min.), cm	15	..

(*c*) That portion of asphalt mastic, when designated as acid resisting, which is insoluble in benzol shall conform to the following requirement:

Matter soluble in diluted HCl....not more than 5 per cent

Methods of Testing

5. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials:

(*a*) *Asphalt Mastic.*—Standard Methods of Testing Bituminous Mastics, Grouts, and Like Mixtures (A.S.T.M. Designation: D 147).[3]

(*b*) *Softening Point.*—Standard Method of Test for Softening Point of Bituminous Materials (Ring-and-Ball Method) (A.S.T.M. Designation: D 36).[3]

(*c*) *Penetration.*—Standard Method of Test for Penetration of Bituminous Materials (A.S.T.M. Designation: D 5).[3]

(*d*) *Ductility.*—Standard Method of Test for Ductility of Bituminous Materials (A.S.T.M. Designation: D 113).[3]

(*e*) *Loss on Heating.*—Tentative Method of Test for Loss on Heating of Oil and Asphaltic Compounds (A.S.T.M. Designation: D 6).[3]

(*f*) *Sieve Analysis.*—Standard Method of Test for Sieve Analysis of Fine and Coarse Aggregates (A.S.T.M. Designation: C 136).[3]

(*g*) *Recovery and Preparation of Matter Soluble in Benzol.*—Until an A.S.T.M. method is available, recovery and preparation for test of the bituminous matter soluble in pure benzol shall be made according to the "Method and Apparatus for the Recovery of Asphalt," as described in the paper by Gene Abson.[4]

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[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 704 (1933).

Standard Specifications for

BITUMINOUS GROUT FOR USE IN WATERPROOFING ABOVE GROUND LEVEL[1]

A.S.T.M. Designation: D 170 - 41

ADOPTED, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 170; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. (*a*) These specifications cover the materials for bituminous grout suitable for use in waterproofing above ground level, either as a protective covering of membrane systems of waterproofing, or for bedding brick or filling the joints or flooding the surface of a brick protective covering.

(*b*) The grade of bituminous grout covered by these specifications is suitable for waterproofing railroad bridges, culverts, subways, etc.

Properties

2. The bituminous grout shall consist of a mixture of substantially 45 parts by weight of bituminous binder and 55 parts by weight of mineral aggregate as coarse as sand, which mixture becomes sufficiently fluid when heated to approximately 205 C. (400 F.), to flow without mechanical manipulation, and which on cooling congeals to a compact mass.

Bituminous Binder

3. The bituminous binder shall consist of either asphalt or coal-tar pitch as follows:

(*a*) *Asphalt.*—The asphalt shall conform to the requirements for type B asphalt as prescribed in the Tentative Specifications for Asphalt for Dampproofing and Waterproofing (A.S.T.M. Designation: D 449) of the American Society for Testing Materials.[3]

(*b*) *Coal-Tar Pitch.*—The coal-tar pitch shall conform to the requirements for type A pitch as prescribed in the Standard Specifications for Coal-Tar Pitch for Roofing, Dampproofing, and Waterproofing (A.S.T.M. Designation: D 450) of the American Society for Testing Materials.[3]

Mineral Aggregate

4. The mineral aggregate shall con-

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] Prior to their present adoption as standard, these specifications were published as tentative from 1923 to 1925, being revised in 1924. They were adopted in 1925, published as standard from 1925 to 1939, but withdrawn and republished as tentative from 1939 to 1941.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

sist of siliceous sand all of which passes a No. 20 (840-micron) sieve, and not more than 5 per cent of which passes a No. 200 (74-micron) sieve.

Methods of Testing

5. The properties enumerated in Sections 2 and 4 shall be determined in accordance with the Standard Methods of Testing Bituminous Mastics, Grouts, and Like Mixtures (A.S.T.M. Designation: D 147) of the American Society for Testing Materials.[3]

Standard Specifications for

BITUMINOUS GROUT FOR USE IN WATERPROOFING BELOW GROUND LEVEL[1]

A.S.T.M. Designation: D 171 - 41

ADOPTED, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 171; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. (*a*) These specifications cover the materials for bituminous grout suitable for use in waterproofing below ground level, either as a protective covering of membrane systems of waterproofing, or for bedding brick or filling the joints or flooding the surface of a brick protective covering.

(*b*) The grade of bituminous grout covered by these specifications is suitable for waterproofing tunnels, subways, etc.

Properties

2. The bituminous grout shall consist of a mixture of substantially 45 parts by weight of bituminous binder and 55 parts by weight of mineral aggregate as coarse as sand, which mixture becomes sufficiently fluid when heated to approximately 149 C. (300 F.), to flow without mechanical manipulation, and which on cooling congeals to a compact mass.

Bituminous Binder

3. The bituminous binder shall consist of either asphalt or coal-tar pitch as follows:

(*a*) *Asphalt.*—The asphalt shall conform to the requirements for type A asphalt as prescribed in the Tentative Specifications for Asphalt for Dampproofing and Waterproofing (A.S.T.M. Designation: D 449) of the American Society for Testing Materials.[3]

(*b*) *Coal-Tar Pitch.*—The coal-tar pitch shall conform to the requirements for type B pitch as prescribed in the Standard Specifications for Coal-Tar Pitch for Roofing, Dampproofing, and Waterproofing (A.S.T.M. Designation: D 450) of the American Society for Testing Materials.[3]

Mineral Aggregate

4. The mineral aggregate shall con-

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] Prior to their present adoption as standard, these specifications were published as tentative from 1923 to 1925, being revised in 1924. They were adopted in 1925, published as standard from 1925 to 1939, but withdrawn and republished as tentative from 1939 to 1941.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

sist of siliceous sand all of which passes a No. 20 (840-micron) sieve, and not more than 5 per cent of which passes a No. 200 (74-micron) sieve.

Methods of Testing

5. The properties enumerated in Sections 2 and 4 shall be determined in accordance with the Standard Methods of Testing Bituminous Mastics, Grouts, and Like Mixtures (A.S.T.M. Designation: D 147) of the American Society for Testing Materials.[3]

Standard Specifications for

ASPHALT-SATURATED ROOFING FELT FOR USE IN WATERPROOFING AND IN CONSTRUCTING BUILT-UP ROOFS[1]

A.S.T.M. Designation: D 226 – 44

ADOPTED, 1942; REVISED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 226; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover asphalt-saturated felts, either 36 or 32 in. in width, composed of roofing felt saturated, but not coated, with asphalt for use in the membrane system of waterproofing and in the construction of built-up roofs.

Types

2. Asphalt-saturated felts covered by these specifications are of two types: namely,

15-lb. type
30-lb. type

Mopping Materials

3. (*a*) For the construction of built-up roofs, mopping asphalts conforming to the Standard Specifications for Asphalt for Use in Constructing Built-Up Roof Coverings (A.S.T.M. Designation: D 312)[3] shall be used.

(*b*) For the construction of a membrane system of waterproofing, types A, B, or C mopping asphalt conforming to the Tentative Specifications for Asphalt for Dampproofing and Waterproofing (A.S.T.M. Designation: D 449)[3] shall be used depending upon service conditions as described in Section 4 of Specifications D 449.

Manufacture

4. In the process of manufacture, a single thickness of dry roofing felt shall be saturated with an asphaltic saturant.

Character of Felt

5. The felt shall be a roofing felt produced by "felting" vegetable or animal fibers or mixtures thereof. The surface of the felt shall be uniformly smooth.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] Prior to their present adoption as standard, these specifications were published as tentative from 1925 to 1927, being revised in 1926 and 1927. They were adopted in 1927, published as standard from 1927 to 1938, but withdrawn, revised, and republished as tentative in 1938, being revised in 1941 and 1942.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Upon splitting or tearing on the bias, the felt shall appear reasonably free from lumps of underbeaten stock (that is, stock that has not been beaten or shredded into fiber in the process of manufacture) and particles of foreign substances (that is, fragments of stone, metal, leather, rubber, etc.).

Character of Saturant

6. The bitumen of the saturant shall be composed of asphaltic materials.

Character of Saturated Felt

7. The felt shall be thoroughly and uniformly saturated, and shall show no unsaturated spots at any point upon cutting 2-in. strips at random across the entire sheet and splitting them open for their full length.

Surface Finish

8. The surface of the felt shall not be coated or covered with talc or other substance that would tend to interfere with the adhesion between the felt and the plying cement.

Physical Properties

9. The fabricated product shall conform to the following requirements:

	15-lb. Type	30-lb. Type
Width of roll	either 36 or 32 in. as may be specified, ±¼ in.	
Area of roll, min., sq. ft.	432	216
Minimum weight of saturated felt per 100 sq. ft. (exclusive of wrapping and packing material), lb.	14	28
Loss on heating at 105° C. (221 F.) for 5 hr., max., per cent		4
Pliability at 77 F. (25 C.): The 10 strips tested shall not crack when bent 90 deg. at a uniform speed over a rounded corner of	½ in. rad.	¾ in. rad.
Average breaking strength at 77 F. (25° C.):		
With fiber grain, min., lb.	30	40
Across fiber grain, min., lb.	15	20
Saturant in moisture-free felt, min., per cent by weight	140	150

Desaturated Felt

10. The desaturated moisture-free felt shall conform to the following requirements:

	15-lb. Type	30-lb. Type
Weight per 100 sq. ft., min., lb	5.2	10.0
Ash, max., per cent	10	10

Freedom from Defects

11. The finished material shall be free of visible external defects, such as holes, ragged or untrue edges, breaks, cracks, tears, protuberances, and indentations.

Pliability and Stickiness

12. The rolls shall not crack nor be so sticky as to cause tearing or material damage upon being unrolled at atmospheric temperatures above 50 F. (10 C.).

Packing

13. The rolls of saturated felt need not be wound on cores, but they shall be securely wrapped in a substantial grade of paper of the same width as the fabric. The wrapper shall completely encircle the roll and shall be pasted at the overlap in a manner that will prevent it shifting from position. The ends of the roll need not be covered. As an alternative, the rolls of saturated felt may either be tied with strings or simply pasted at the ends, as may be agreed upon by the purchaser and the seller. No roll shall contain more than two pieces, and there shall not be more than 3 per cent of rolls containing two pieces in any shipment.

Marking

14. Each roll shall be plainly marked with the manufacturer's name and

brand or as agreed upon by the purchaser and the seller, and shall bear a notation showing the type of saturant.

Sampling and Testing

15. The material shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the Tentative Methods of Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing and Roofing (A.S.T.M. Designation: D 146).[3]

Inspection

16. Inspection of material shall be made as mutually agreed upon by the purchaser and the seller as part of the purchase contract.

Basis of Rejection

17. Failure to conform to any one of the requirements prescribed in these specifications shall constitute grounds for rejection. In case of rejection, the seller shall have the right to reinspect the rejected shipment and resubmit the lot after removal of those packages not conforming to the specified requirements.

Standard Specifications for

COAL-TAR SATURATED ROOFING FELT FOR USE IN WATERPROOFING AND IN CONSTRUCTING BUILT-UP ROOFS[1]

A.S.T.M. Designation: D 227 – 44

ADOPTED, 1927; REVISED, 1941, 1942, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 227; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover coal-tar saturated felt, either 36 or 32 in. in width, composed of roofing felt saturated, but not coated, with refined coal tar, for use in the membrane system of waterproofing and in the construction of built-up roofs.

Mopping Materials

2. (*a*) For the construction of built-up roofs on inclines not exceeding 3 in. per ft. where nailing is employed and not exceeding 1 in. per ft. where nailing is omitted, type A coal-tar pitch conforming to the Standard Specifications for Coal-Tar Pitch for Roofing, Dampproofing, and Waterproofing (A.S.T.M. Designation: D 450)[3] shall be used.

(*b*) For the construction of steep built-up roofs with inclines of 1 to 6 in. per horizontal foot where nailing is employed, the Tentative Specifications for Coal-Tar Pitch for Steep Built-Up Roofs (A.S.T.M. Designation: D 654)[3] may be used.

(*c*) For the construction of a membrane system of waterproofing, types A or B coal-tar pitch conforming to the Standard Specifications for Coal-Tar Pitch for Roofing, Dampproofing, and Waterproofing (A.S.T.M. Designation: D 450)[3] shall be used depending upon the conditions of service as described in Section 4 of Specifications D 450.

Manufacture

3. In the process of manufacture, a single thickness of dry roofing felt shall be saturated with a coal-tar saturant.

Character of Felt

4. The felt shall be a roofing felt produced by "felting" vegetable and animal fibers or mixtures thereof. The surface

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] Prior to adoption as standard, these specifications were published as tentative from 1925 to 1927, being revised in 1926 and 1927.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

of the felt shall be uniformly smooth. Upon splitting or tearing on the bias, the felt shall appear reasonably free from lumps of underbeaten stock (that is, stock that has not been beaten or shredded into fiber in the process of manufacture) and particles of foreign substances (that is, fragments of stone, metal, leather, rubber, etc.).

Character of Saturant

5. The saturant shall be composed of coal tar from which the highly volatile constituents have been removed.

Character of Saturated Felt

6. The felt shall be thoroughly and uniformly saturated and shall show no unsaturated spots at any point upon cutting 2-in. strips at random across the entire sheet and splitting them open for their full length.

Surface Finish

7. The surface of the felt shall not be coated or covered with talc or other substance that would tend to interfere with the adhesion between the felt and the plying cement.

Physical Properties

8. The fabricated product shall conform to the following requirements:

Width of roll	either 36 or 32 in., as may be specified, ± ¼ in.
Area of roll	432 sq. ft., min.
Weight of material per 100 sq. ft. exclusive of packing and wrapping	14 lb., min.
Moisture	2.5 per cent of the net weight, max.
Average breaking strength at 77 F. (25 C.):	
With fiber grain	30 lb., min.[a]
Across fiber grain	15 lb., min.[a]
Pliability at 77 F. (25 C.)	the 10 strips tested shall not crack when bent 90 deg. at a uniform speed over a rounded corner of ½-in. radius
Saturant in moisture-free felt	140 per cent by weight, min.

[a] If samples fail to conform to the requirements for breaking strength immediately after sampling, a retest shall be made upon test specimens which have been suspended in free air at laboratory temperature for 72 hr. Material shall be accepted or rejected upon the basis of this retest.

Desaturated Felt

9. The desaturated moisture-free felt shall conform to the following requirements:

Weight per 100 sq. ft., min., lb	5.2
Ash, max., per cent	10

Freedom from Defects

10. The finished material shall be free of visible external defects, such as holes, ragged or untrue edges, breaks, cracks, tears, protuberances, and indentations.

Pliability and Stickiness

11. The rolls shall not crack, nor be so sticky as to cause tearing or material damage upon being unrolled at atmospheric temperatures above 50 F. (10 C.).

Packing

12. The rolls of saturated felt need not be wound on cores, but they shall be securely wrapped in a substantial grade of paper of the same width as the fabric. The wrapper shall completely encircle the roll and shall be pasted at the overlap in a manner that will prevent it shifting from position. The ends of the roll need not be covered. As an alternative, the rolls of saturated felt may either be tied with strings or simply pasted at the ends, as may be agreed upon by the purchaser and the seller. No roll shall contain more than two pieces, and there shall not be more than 3 per cent of rolls containing two pieces in any shipment.

Marking

13. Each roll shall be plainly marked with the manufacturer's name and brand or as agreed by the purchaser and the seller, and shall bear a notation showing the type of saturant.

Sampling and Testing

14. The material shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the Tentative Methods of Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing and Roofing (A.S.T.M. Designation: D 146).[3]

Inspection

15. Inspection of material shall be made as agreed upon by the purchaser and the seller as part of the purchase contract.

Basis of Rejection

16. Failure to conform to any one of the requirements prescribed in these specifications shall constitute grounds for rejection. In case of rejection, the seller shall have the right to reinspect the rejected shipment and resubmit the lot after removal of those packages not conforming to the specified requirements.

Standard Specifications for WOVEN COTTON FABRICS SATURATED WITH BITUMINOUS SUBSTANCES FOR USE IN WATERPROOFING[1]

A.S.T.M. Designation: D 173 – 44

ADOPTED, 1942; REVISED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 173; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover bituminized cotton fabric, composed of woven cotton cloth waterproofed with either asphalt or coal-tar pitch, as specified by the purchaser, for use in the membrane system of waterproofing.

Mopping Materials

2. (*a*) For the construction of a membrane system of waterproofing with cotton fabric saturated with asphalt, types A, B, or C asphalt conforming to the Tentative Specifications for Asphalt for Dampproofing and Waterproofing (A.S.T.M. Designation: D 449)[3] shall be used depending upon the conditions of service as described in Section 4 of Specifications D 449.

(*b*) For the construction of a membrane system of waterproofing with woven cotton fabric saturated with coal-tar, coal-tar pitch conforming to the Standard Specifications for Coal-Tar Pitch for Roofing, Dampproofing, and Waterproofing (A.S.T.M. Designation: D 450)[3] shall be used.

Manufacture

3. In the process of manufacture, the dry cotton fabric shall be thoroughly and uniformly waterproofed with either an asphaltic or coal-tar pitch saturant, at a temperature and speed that will not injure the fabric. This shall be accomplished either by passing the fabric through the saturant or by spraying with saturant and then calendering it in the presence of heat, whereupon it shall be cooled and wound into rolls.

Character of Saturated Fabric

4. The meshes of the fabric shall not be completely closed or sealed by the process of saturation, but there shall be sufficient porosity maintained to allow

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] Prior to their present adoption as standard these specifications were published as tentative from 1923 to 1925, being revised in 1925. They were adopted in 1925, published as standard from 1925 to 1938, being revised in 1927, but withdrawn, revised and republished as tentative in 1938, being revised in 1939 and 1940.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

successive moppings of the plying cement to seep through.

Surface Finish

5. (*a*) The surface of the fabric shall not be coated or covered with talc or other substance that would tend to interfere with the adhesion between the fabric and the plying cement. The use of silica or wood flour will be permitted.

(*b*) The surface shall be uniformly smooth and free from irregularities, folds, or knots.

Selvage

8. If selvage is used, it shall measure not more than $\frac{1}{4}$ in.

Freedom from Defects

9. The finished material shall be free of visible external defects such as ragged or untrue edges, breaks, rents, or cracks.

Pliability and Stickiness

10. The roll shall be capable of being unrolled easily at atmospheric temperatures above 50 F. (10 C.) without sticking together in such a manner as to injure the fabric.

TABLE I.—PHYSICAL REQUIREMENTS FOR SATURATED COTTON FABRICS FOR WATERPROOFING.

	Min.	Max.
Width of roll	30 in.	38 in.
Gross weight per roll	35 lb.	80 lb.
Weight of wrapping material and mandrel per roll	.....	2.5 lb.
Average net weight per square yard	10 oz.	.....
Detached comminuted surfacing per 100 sq. ft.	.....	1 lb.
Moisture content based on net weight	.....	4 per cent
Average strength at 70 F. (21.1 C.) measured lengthwise of the roll (that is, in the direction of the warp)	50 lb.[a]	.....
Average strength at 70 F. (21.1 C.) measured crosswise of the roll (that is, in the direction of the filling)	50 lb.[a]	.....
Average loss on heating (exclusive of moisture) of asphalt-saturated fabrics	.....	4 per cent
Saturant in fabricated material	1.6 times the weight of the moisture-free fabric in the same area	.....
Pliability at 32 F. (0 C.)	No cracking of the fabricated bituminized fabric shall take place on bending over a ½-in. mandrel through an arc of 180 deg. in one direction and then through 360 deg. over the same mandrel in the opposite direction	

[a] The committee recognizes the advisability of including a requirement relative to the percentage elongation before rupture, but in the absence of any reliable method of test, such requirement cannot be stated at the present time.

Physical Properties

6. The fabricated product shall conform to the requirements prescribed in Table I.

Desaturated Fabric

7. The desaturated woven cotton fabric shall conform to the following requirements:

Average dry weight per square yard, min	3½ oz.
Ash (based on dry weight of fabric), max	2 per cent.
Composition of fabric	not less than 100 per cent[a] of cotton fibers
Thread count per inch:	
Warp	26 to 32
Filling	24 to 32

[a] This shall not exclude the presence of the customary sizing materials in amounts not exceeding 8 per cent by weight of the moisture-free cotton.

Packing

11. The rolls of treated cotton fabric shall be wound on mandrels or rigid hollow fiber cylinders not less than 2 in. in diameter or 2 in. square, extending not more than 4 in. beyond the ends of the rolls. They shall be securely tied or wrapped in a manner that will prevent unrolling in transit.

Sampling and Testing

12. The material shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the Tentative Methods

of Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing and Roofing (A.S.T.M. Designation: D 146).[3]

Inspection

13. Inspection of material shall be made as agreed upon by the purchaser and the seller as part of the purchase contract.

Basis of Rejection

14. Failure to conform to any one of the requirements prescribed in these specifications shall constitute grounds for rejection. In case of rejection, the seller shall have the right to reinspect the rejected shipment and resubmit the lot after removal of those packages not conforming to the specified requirements.

Standard Specifications for

WIDE SELVAGE ASPHALT ROOFING SURFACED WITH COARSE MINERAL GRANULES[1]

A.S.T.M. Designation: D 371 - 44

ADOPTED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 371; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover asphalt roofing in sheet form, either 36 or 32 in. in width, composed of asphalt-saturated roofing felt coated on approximately one half of the width of the weather side with asphalt, and surfaced on the coated portion with mineral granules. This roofing is used as a cap sheet in the construction of built-up roofs.

Manufacture

2. In the process of manufacture, a single thickness of dry roofing felt shall be impregnated with a hot asphaltic saturant, then coated on the weather side for approximately one half the width of the sheet with an asphaltic coating (compounded with a fine mineral filler substantially insoluble in water), and surfaced by embedding mineral granules in the hot asphaltic coating. The coated and surfaced portion shall be 17 in. in width on 36-in. roofing and 15 in. in width on 32-in. roofing.

Character of Felt

3. The felt shall be a roofing felt produced by "felting" vegetable or animal fibers or mixtures thereof. The surface of the felt shall be uniformly smooth. Upon splitting or tearing on the bias, the felt shall appear reasonably free from lumps of underbeaten stock and particles of foreign substances (that is, fragments of stone, metal, leather, rubber, etc.).

Character of Saturant and Coatings

4. The bitumen of the saturant and coatings shall be composed of asphaltic materials.

Character of Saturated Felt

5. The felt shall be thoroughly and uniformly saturated, and shall show no unsaturated spots at any point upon

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] Prior to adoption as standard, these specifications were published as tentative from 1933 to 1944, being revised in 1937, 1941, and 1944.

cutting 2-in. strips at random across the entire sheet and splitting them open for their full length.

Surface Finish

6. (*a*) The surface of the weather side shall be uniform in finish and texture. The mineral granules on the surfaced portion shall be uniformly distributed in a smooth layer and shall be firmly embedded in the asphalt coating.

(*b*) The line between the surfaced and unsurfaced portion shall be straight and shall run parallel longitudinally to the edge of the sheet. The surfaced portion shall be 17 ± ½ in. on 36-in. roofing and 15 ± ½ in. on 32-in. roofing.

(*c*) The reverse side of the sheet may or may not be coated. If coated, it may be surfaced with a small amount of mineral matter to prevent sticking in the roll.

Physical Properties

7. The fabricated product shall conform to the requirements prescribed in Table I.

Freedom from Defects

8. The finished material shall be free of visible external defects, such as holes, ragged or untrue edges, breaks, cracks, tears, protuberances, and indentations.

Pliability and Stickiness

9. The finished product shall not crack nor be so sticky as to cause tearing or material damage upon being unrolled at atmospheric temperatures above 50 F. (10 C.).

Packing

10. (*a*) Cap sheets shall be put up in rolls, containing sufficient material to cover not less than 50 sq. ft. No roll shall contain more than two pieces nor shall there be more than 3 per cent of rolls containing two pieces in any shipment. The rolls shall be tightly wound, and shall be securely wrapped in a substantial grade of paper completely

TABLE I.—PHYSICAL REQUIREMENTS FOR ASPHALT CAP SHEET.

Width	The roofing shall be put up in rolls and shall be either 36 or 32 in. in width, as may be specified, within a permissible variation of ±¼ in.			
Area	The average area of the rolls examined shall contain sufficient roofing to cover 50 sq. ft. of roof surface: for roofing 32 in. in width, 107.3 sq. ft. ± 5 per cent, and for roofing 36 in. in width, 106.8 sq. ft. ± 5 per cent.			
Pliability at 77 F. (25 C.)[a]	At least 8 strips out of 10 from the granule-surfaced portion shall not crack when bent 90 deg. at a uniform speed over a rounded corner of ¾-in. radius.			
Behavior on heating to 176 F. (80 C.) for 2 hr.[a]	Not more than 1.5 per cent volatile matter loss. The granular surfacing shall not slide more than ¼ in. when suspended vertically.			
	45-lb. Grade		55-lb. Grade	
	Max., lb.	Min., lb.	Max., lb.	Min., lb.
Weight per roll, net average	....	42	. . .	52
Weight of dry felt per 108 sq. ft.	....	7.1	. . .	10.8
Weight of saturant (soluble in CS_2) per 108 sq. ft.[b]	....	10.0	....	17.5
Weight of weather side mineral filled coating per 108 sq. ft.[a]	35.0	16.0	35.0	16.0
Weight of weather side mineral matter passing No. 6 (3360-micron) and retained on No. 100 (149-micron) sieve per 108 sq. ft.[a]	....	20.0	....	20.0
Percentage by weight of mineral matter passing No. 100 (149-micron) sieve on the basis of the sum of the bitumen in the coatings and the mineral matter passing No. 100 (149-micron) sieve	50.0 per cent	15.0 per cent	50.0 per cent	15.0 per cent
Weight of any 108 sq. ft. roll of roofing in shipment	....	40.0	....	50.0

[a] Granule-surfaced portion.

[b] The weight of saturant per 108 sq. ft. shall be not less than 1.4 times the weight of the dry felt on the 45-lb. grade and 1.6 times the weight of the dry felt on the 55-lb. grade.

encircling the roll and pasted at the overlap in such a manner as to prevent shifting from position.

(*b*) Special packing shall be as agreed upon by the purchaser and the seller.

Marking

11. Each roll shall be plainly marked with the manufacturer's name and brand.

Sampling and Testing

12. The material shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the Tentative Methods of Testing Asphalt Roll Roofing, Cap Sheets, and Shingles (A.S.T.M. Designation: D 228) of the American Society of Testing Materials.[3]

Inspection

13. Inspection of material shall be made as agreed upon by the purchaser and the seller as part of the purchase contract.

Basis of Rejection

14. Failure to conform to any one of the requirements prescribed in these specifications shall constitute grounds for rejection. In case of rejection, the seller shall have the right to reinspect the rejected shipment and resubmit the lot after removal of those packages not conforming to the specified requirements.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for

ASPHALT SIDING SURFACED WITH COARSE MINERAL GRANULES[1]

A.S.T.M. Designation: D 699 – 44

ADOPTED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 699; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. (*a*) These specifications cover asphalt siding in strip or individual form surfaced with colored mineral granules, composed of roofing felt saturated and coated on both sides with asphalt and surfaced on the weather side with granulated slate or equivalent mineral material (of solid or mixed colors as may be agreed upon by the purchaser and the seller), and on the reverse side with a surfacing material.

(*b*) Siding shingles supplied under these specifications are intended to be used with a "headlap" (Note) of not less than 1 in.

NOTE.—The headlap is the shortest vertical distance between the top of cutout or bottom edge of a course of siding and the most proximate underlying area of sidewall not covered by preceding courses of siding.

Manufacture

2. In the process of manufacture, a single thickness of dry roofing felt shall be impregnated with a hot asphaltic saturant, then coated on both sides with a hot asphaltic coating compounded with a fine mineral filler substantially insoluble in water, and finally surfaced on the weather side with mineral granules embedded in the hot asphaltic coating. The reverse side shall be covered with a suitable material to prevent sticking in the package.

Character of Felt

3. The surface of the felt shall be uniformly smooth. Upon splitting or tearing on the bias, the felt shall appear reasonably free from lumps of underbeaten stock and particles of foreign substances (that is, fragments of stone, metal, leather, rubber, etc.)

Character of Saturant and Coatings

4. The bitumen of the saturant and coatings shall be composed of asphaltic materials.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] Prior to adoption as standard, these specifications were published as tentative from 1942 to 1944, being revised in 1944.

Character of Saturated Felt

5. The felt shall be completely and uniformly saturated, and shall show no unsaturated spots at any point upon cutting 2-in. strips at random across the entire piece and splitting them open for their full length.

Surface Finish

6. (*a*) The surface of the weather side shall be uniform in finish and may be embossed to simulate a grained texture. The mineral granules shall cover the entire surface and shall be firmly embedded in the asphalt coating.

(*b*) The reverse side shall have a coating of asphalt and shall be surfaced with suitable material to prevent sticking.

Physical Properties

7. The fabricated product shall conform to the requirements prescribed in Table I.

Freedom from Defects

8. The finished material shall be free of visible external defects, such as holes, ragged or untrue edges, rents, cracks, indentations, and lumps of coating.

Stickiness

9. The siding shall not stick together in the packages so as to cause damage upon being unpacked at atmospheric temperatures above 50 F. (10 C.).

Packing

10. The siding shall be packed in cardboard cartons or between wood or cardboard ends and tied securely so as to make a substantial package weighing not more than 135 lb., gross.

Marking

11. Each package shall be plainly marked with the name and brand of the manufacturer, as well as the style, type, and color of the finished product. Direction sheets for application shall be included in at least every fifth package.

Sampling and Testing

12. The material shall be sampled and the properties enumerated in these specifications shall be determined in ac-

TABLE I.—PHYSICAL REQUIREMENTS OF ASPHALT SIDING.

Shape	The form and size of the shingles shall be as agreed upon between the purchaser and the seller.		
Area	Sufficient material per "square" shall be furnished to adequately waterproof 100 sq. ft. of sidewall area.		
Behavior on heating to 176 F. (80 C.) for 2 hr	Not more than 1.5 per cent volatile matter loss. The granular surfacing shall not slide more than 1/8 in. when suspended vertically.		
	Regular Weight, lb.		
	Max.	Min.	
Weight, net average per 100 sq. ft.[a]	...	75	
Weight of dry felt per 100 sq. ft.	...	10.0	
Weight of saturant per 100 sq. ft.[b]	...	17.5	
Weight of weatherside mineral filled coating per 100 sq. ft.	...	10	
Weight of mineral matter per 100 sq. ft. passing No. 6 (3360-micron) and retained on No. 100 (149-micron) sieve	...	18.5	
Percentage by weight of mineral matter passing No. 100 (149-micron) sieve on the basis of the sum of the bitumen in the coatings and the mineral matter passing No. 100 (149-micron) sieve	50	15	
Weight of siding per 100 sq. ft. (individual package)	...	72	

[a] Weight, net average per 100 sq. ft. $= \frac{\text{net wt. of packages inspected}}{\text{sq. ft. area per siding piece} \times \text{no. of pieces}} \times 100$.

[b] The weight of saturant per 100 sq. ft. shall be not less than 1.75 times the weight of the dry felt.

cordance with the Tentative Methods of Testing Asphalt Roll Roofing, Cap Sheets, and Shingles (A.S.T.M. Designation: D 228) of the American Society for Testing Materials.[3]

Inspection

13. Inspection of material shall be made as agreed upon by the purchaser and the seller as part of the purchase contract.

Basis of Rejection

14. Failure to conform to any one of the requirements prescribed in these specifications shall constitute grounds for rejection. In case of rejection, the seller shall have the right to reinspect the rejected shipment and resubmit the lot after removal of those packages not conforming to the specified requirements.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Methods of

TESTING BITUMINOUS MASTICS, GROUTS, AND LIKE MIXTURES[1]

A.S.T.M. Designation: D 147 – 41

ADOPTED, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 147; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods cover the procedures for testing bituminous mastics, grouts, and like mixtures, which may be classified and defined as follows:

Bituminous Grout.—A mixture of bituminous material as a binder and sandy mineral matter as an aggregate, which when heated to a suitable temperature becomes sufficiently fluid to flow into place without mechanical manipulation, and which on cooling congeals to a compact mass.

Asphalt Mastic.—A mixture containing (1) asphaltic material as a binder and graded mineral matter as an aggregate, or (2) pulverized native rock asphalt (to which asphaltic material may have been added); either of which when heated to a suitable temperature may be poured into place but which requires trowelling to form it into a compact mass.

Asphalt Mastic Cake.—A mixture containing asphaltic material as a binder and an aggregate consisting chiefly of calcareous or siliceous dust cast into the form of blocks or "cakes" and adapted for use in preparing asphalt mastic.

Preparation of Samples

2. (*a*) Bituminous grouts shall be heated in an oven or on a hot plate in a pan or other suitable container at the lowest possible temperature to prevent overheating and volatilization, and, when sufficiently fluid, shall be thoroughly stirred to insure a uniform sample, whereupon 10 to 30 g. shall be taken for analysis.

(*b*) Asphalt mastics or mastic cake shall be warmed on a hot plate or in a hot oven until soft enough to be broken up or stirred so that a representative sample for analysis may be taken. The amount taken for analysis will depend upon the amount of coarse gravel or stone in the mixture. The larger the

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] Prior to their present adoption as standard, these methods were published as tentative from 1922 to 1927, being revised in 1923, 1924, 1925, and 1927. They were adopted in 1927, published as standard from 1927 to 1939, but withdrawn, revised, and republished as tentative from 1939 to 1941.

Emergency Alternate Provisions EA – D 147 applying to this standard appear in the pink section of this book.

gravel or stone, the larger will be the sample required for accuracy. The size of samples to be taken shall be as follows: Where all particles pass a No. 10 (2000-micron) sieve, 10 to 30 g.; where 25 per cent of the aggregate is retained on a No. 10 sieve, 50 g.; where 50 per cent of the aggregate is retained on a No. 10 sieve, 100 g.; and where 75 per cent of the aggregate is retained on a No. 10 sieve, 200 g.

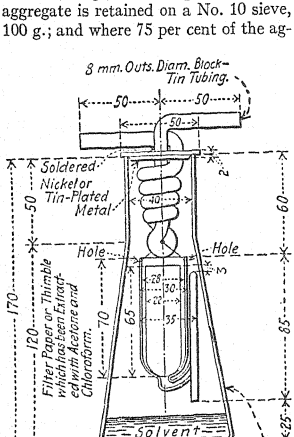


FIG. 1.—Extraction Apparatus for Analysis of 10 to 30-g. Samples.

NOTE.—Where the properties of the extracted bituminous matter are to be determined, 500 g. shall be taken and extracted with pure benzol as described in Sections 3 (*b*) and 5 and examined as specified in Section 6.

Apparatus

3. (*a*) *For Analysis of 10 to 30-g. Samples.*—In cases where a 10 to 30-g. sample is sufficient, the analysis shall be carried out by means of the glass extractor, shown in Fig. 1.

(*b*) *For Analysis of 50 to 500-g. Samples.*—The apparatus for analysis of samples containing coarser aggregate shall be the large extractor, shown in Fig. 2, consisting of a large brass cylinder, through the bottom of which projects a 16-candlepower, incandescent carbon filament bulb or other type of electric heater of correct capacity to supply heat to the extraction apparatus proper, held in the upper portion of the cylinder. This apparatus shall consist of a cylindrical brass vessel for holding the solvent, a cylindrical wire basket made of 80-mesh wire cloth suspended in the cylinder, and an inverted conical condenser which serves as a top.

Procedure for Analysis of 10 to 30-g. Samples[3]

4. (*a*) An ordinary Whatman or S. and S. filter thimble shall be dried and weighed. The weighed sample shall be placed in the thimble and 40 to 50 ml. of CS_2[4] poured over the sample. The thimble containing the sample shall be suspended under the condenser by a fine wire bail as shown in Fig. 1. The flask shall be cautiously heated by a steam-bath or electric heater just enough to vaporize the solvent. Cold water shall be circulated through the condenser. The heat evaporates the carbon disulfide in the flask. This condenses upon the condenser and drops back upon the sample through which it filters, thus dissolving out the bitumen which collects in the bottom of the flask. The extraction should be discontinued when the

[3] This method for analysis of larger samples is especially adapted for asphalt mastics, grouts, and mastic cake. If the binder is coal-tar pitch, the "free carbon" constituent of the binder will remain with the mineral aggregate. The committee has under consideration a possible separate method for analysis of coal-tar mastics.

[4] Carbon tetrachloride, benzol, or chloroform may be used instead of carbon disulfide, with the only difference that in the case of noninflammable solvents, the solvent will have to be evaporated from the solution of bitumen to determine the ash for correction instead of burning off directly. When the solvent is expelled, the bitumen can be ignited for ash.

CS_2 drops colorless from the filter. The time of extraction will depend upon the nature of the bitumen and mineral aggregate in the sample and upon the degree of heat applied, the coldness of the water in the condenser, and other factors. In some cases extraction may be complete in 1 hr., in others 4 or 5 hr. may be necessary. When the solvent comes through clear, the filter shall be removed and washed with a fine jet of CS_2 from a washing bottle to wash out any bitumen that may be retained at the top of the paper and to break up any channels that may have been formed by the CS_2 passing through. If the washings show any color, the thimble shall be put back and extraction continued until the solvent again becomes colorless. It shall then be removed, dried carefully, at a low temperature at first to prevent ignition of the absorbed CS_2, and finally to constant weight at 100 C. (212 F.), cooled, and weighed. The solution in the flask shall be rinsed into a weighed porcelain or silica evaporating dish or crucible and the solvent burned off under a hood. The residue shall be ignited over a flame or in a muffle and the ash weighed and the weight added to that of the mineral matter in the filter paper. This is to correct for the fine mineral matter which will be carried through the paper by the solvent. Should there be a considerable amount of ash recovered in this way, and if it is found that the mineral matter is calcium or other carbonate, it shall be recarbonated by repeated treatment with ammonium carbonate solution and finally ignited at a dull red heat. Ordinarily, however, the mineral matter going through the paper will be so small in amount that the difference caused by ignition may be neglected. The corrected loss in weight on the original sample represents the percentage of bitumen.

(*b*) The sieve analysis of the mineral aggregate shall be made in accordance with the Standard Method of Test for Sieve Analysis of Fine and Coarse Aggregates (A.S.T.M. Designation: C 136) of the American Society for Testing Materials.[5] The extracted residue shall be transferred from the thimble to the No. 200 (74-micron) sieve, the paper being gently rubbed to free adhering particles. The aggregate on the sieve shall be gently rubbed with the fingers to break up lumps and to free any particles of fine dust that might adhere to larger sand particles. The sieve shall be shaken over a piece of paper from side to side with the right hand, striking it sharply against the palm of the left hand until no appreciable amount of dust comes through the sieve on to the paper. The paper shall be cleared from time to time by raising one side with the left hand and rolling off the siftings, so that it can be seen when the sifting is complete. The material remaining on the No. 200 sieve shall be weighed and the amount of material which has passed through the No. 200 sieve shall be determined by difference. This operation shall be repeated upon the coarser sieves in order and the amount passing each sieve and retained on the next finer recorded as percentage of the original sample.

Procedure for Analysis of 50 to 500-g. Samples

5. (*a*) A large filter paper, 12 or 13 in. in diameter, shall be fitted inside the wire basket of the extractor, shown in Fig. 2, by folding once more than in ordinary filtering, or by wrapping it over a form which fits inside the basket (a cylindrical bottle of proper size makes a good form) and placing it inside the basket. The basket with contained filter paper shall be dried and weighed.

[5] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

The sample shall be weighed and packed in the filter paper in the basket. Care should be taken not to pack all coarse particles in one place and the fine particles in another but to have them mixed together in uniform proportions. The sample shall be covered with a disk of felt or wad of absorbent cotton to insure even distribution of the dropping solvent, thus preventing it from forming a channel through the sample. The basket shall be suspended in the extractor and 150 to 200 ml. of CS_2[4] poured over the felt or cotton. The condenser shall be placed over the top and water circulated through it. Current shall be started through the electric lamp underneath the extractor and the extraction carried on exactly as in the smaller glass extractor, but on a larger scale. The time for extraction will vary from 3 to 12 hr. or more, depending upon the nature of the sample. To determine when extraction is complete, the condenser shall be raised and the basket lifted out to observe if drippings are clear. One or two drops caught upon white filter paper should leave but a light stain.

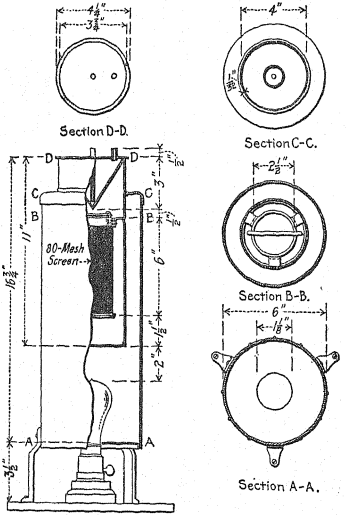


FIG. 2.—Extraction Apparatus for Analysis of 50 to 500-g. Samples.

(*b*) The drying and weighing of the basket, burning off of the solution for correction, and the sifting of the mineral aggregate shall be carried on exactly as in the case of the smaller sample as described in Section 4, except that in sifting mineral aggregates containing coarse stone, it is well to remove the stone by putting the mineral aggregate first through a No. 10 (2000-micron) sieve, as

the large stone would injure the No. 200 (74-micron) sieve.

(*c*) The stone removed by the No. 10 sieve may be sieved through ¼-in., ½-in., and coarser sieves in order, as described in the Standard Method C 136,[5] and the fine material passing the No. 10 sieve through No. 200 and coarser sieves in order, all amounts passing any one sieve and retained on the next finer one being reported as percentage of the original sample. Thus the ingredients of the mixture may be roughly separated by the use of the proper size sieves, and examined for physical and chemical characteristics.

NOTE.—The siftings passing the No. 200 (74-micron) sieve will consist largely of the dust or pulverized rock used in the mixture. The material between the No. 100 (149-micron) and the No. 10 (2000-micron) will consist largely of the sand used in the mixture, with any particles of fine crushed stone within these limits that existed in the original materials. Gravel or broken stone as a rule will be larger than No. 10 or No. 8 (2380-micron).

Recovery and Examination of Extracted Bitumen

6. Until standard methods are available, recovery and preparation for test of the bituminous matter soluble in pure benzol extracted from asphalt mastics and grouts shall be made according to the procedure described in the paper "Method and Apparatus for the Recovery of Asphalt."[6]

[6] Gene Abson, "Method and Apparatus for the Recovery of Asphalt," *Proceedings*, Am. Soc. Testing Mats. Vol. 33, Part II, p. 704 (1933).

Solubility of Aggregate in Hydrochloric Acid

7. When the mastic is to be used for acid-proof floors, tank linings, etc., pulverized silica and other materials insoluble in acid are used in preparing the mixture. The most important chemical property, therefore, is solubility or insolubility in mineral acids. This determination may be carried out in the following manner:

Dry to constant weight at 325 F. about 2.5 g. of the dust, or of the coarser material, or stone which has been previously pulverized in a mortar. The loss will represent any moisture that may have been retained in the material as well as any absorbed solvent that may not have been expelled while drying the sample after extraction. Place 1 g. of the dried sample in a 250-ml. beaker, cover with a watch glass and add 10 ml. of c. p. HCl (sp. gr. 1.19), dilute with 50 ml. of distilled water. Break up any lumps by means of a glass stirring rod. Note any effervescence which may occur. If considerable effervescing takes place, the sample may be said to be carbonate. If all the sample, or practically all, dissolves, the determination need not be carried further. If apparently insoluble, heat it carefully and boil for 15 min., and filter through a tared Gooch filter, and wash the insoluble residue with hot water. Ignite and weigh and report the loss as the percentage soluble in hydrochloric acid.

Standard Method of Test for

COARSE PARTICLES IN MIXTURES OF ASPHALT AND MINERAL MATTER[1]

A.S.T.M. Designation: D 313 – 41

ADOPTED, 1941.[2]

Reapproved in 1944 Without Change.

This Standard of the American Society for Testing Materials is issued under the fixed designation D 313; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test covers the procedure for determining the amount of particles of mineral or other insoluble matter in asphaltic mixtures that are retained upon a No. 200 (74-micron) sieve.

Sampling

2. The material shall be sampled in accordance with the Tentative Methods of Sampling Bituminous Materials (A.S.T.M. Designation: D 140) of the American Society for Testing Materials.[3]

Preparation of Sample

3. The sample as received shall be completely melted in an oven at the lowest possible temperature, stirred well to insure homogeneity, and samples for test taken immediately.

Apparatus

4. (*a*) The apparatus shall consist either of a 3-in. No. 200 (74-micron) sieve or, a crucible sieve, $1\frac{3}{4}$ in. in diameter at the top and $1\frac{1}{4}$ in. in diameter at the bottom containing a No. 200 (74-micron) sieve. The sieve shall conform to the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials.[3]

(*b*) A 3-in. No. 200 (74-micron) sieve for comparison purposes shall be retained in the laboratory as a standard. Whenever a new sieve is secured, a practical test of its accuracy should be made by running on it, and on the standard sieve, a comparison test, using powdered mineral matter that has a considerable percentage of coarse particles. A reserve stock of such powdered mineral filler should be kept for this purpose.

Procedure

5. (*a*) The sieve shall be dried in an oven at 105 to 110 C., cooled, and then weighed on an analytical balance, the weight being recorded to the nearest 1 mg.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] Prior to adoption as standard, these specifications were published as tentative from 1929 to 1941, being revised in 1935 and 1937.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(*b*) A sample of the asphalt sufficient to yield about 1 g. of mineral matter shall be weighed into a 400-ml. beaker. Then carbon disulfide, carbon tetrachloride, or benzol shall be added and the mixture warmed until the asphalt is entirely dissolved. The solution obtained shall be poured through the weighed sieve, and the residue in the beaker shall be washed onto the sieve, using the solvent employed.

(*c*) The sieve containing the residue shall be washed with a fine jet of the solvent from a wash bottle until free from asphalt and fine mineral matter, then dried, tapped lightly to remove any fine mineral matter held on the wires, and weighed. During the washing, the sieve shall be held so that the liquid will drain through readily.

Calculation

6. The percentage of coarse particles in the mineral matter shall be calculated from the weight of the sample taken, the percentage of matter insoluble in carbon disulfide, and the weight of the residue retained on the sieve.

Standard Method of Test for

SIEVE ANALYSIS OF GRANULAR MINERAL SURFACING FOR ASPHALT ROOFING AND SHINGLES[1]

A.S.T.M. Designation: D 451 - 40

ADOPTED, 1940.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 451; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method is intended for the sieve analysis of granular mineral surfacing material, such as crushed slate, stone, etc., used on the weather surface of prepared asphalt roofing and shingles.

Apparatus

2. (*a*) *Sieves.*—A set of consecutive sieves of the series listed in Table I, conforming to the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials,[3] shall be used. The group of sieves selected shall include those appropriate to the grading of the granular mineral to be subjected to sieve analysis. Sieves of either the coarser or finer openings, on which less than 0.05 per cent of the weight of the sample of the particular material would be found after sieving, need not be included in the set. The wire cloth for these sieves shall be woven (not twilled) from brass, bronze, or other suitable wire, and shall be mounted without distortion in circular frames, 8 in. (20.32 cm.) in diameter and about 2 in. (5 cm.) between the top of the frame and the cloth.

(*b*) *Sieve Shaker.*—A mechanically operated sieve shaker, which imparts to the set of sieves a rotary motion and tapping action of uniform speed, shall be used. The number of taps per minute shall be between 140 and 160. The sieve shaker shall be fitted with a hard maple plug to receive the impact of the tapping device. The entire apparatus shall be rigidly mounted by bolting to a solid foundation, preferably of concrete.

(*c*) *Sample Splitter.*—A riffle sampler with $\frac{3}{8}$ or $\frac{1}{2}$-in. divisions, for reducing the gross sample to the quantity required for the sieve analysis, shall be used.

Unit for Sampling

3. Each carload of mineral granules shall be considered the unit for sampling,

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] Prior to adoption as standard, this method was published as tentative from 1937 to 1940.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

except that where a carload includes more than one kind of granular surfacing, the entire quantity of each kind in the car shall be considered the unit for each kind, respectively.

Collection of Gross Sample

4. (*a*) In collecting the gross sample from a carload of mineral granules shipped in bulk, eight equal increments of not less than 2 lb. each shall be taken from the material at the exposed surface of the car. A conical excavation about 1 ft. in depth shall be made at each sampling point and the sample taken from the bottom of the excavation. The eight sampling points shall be located as shown in Fig. 1.

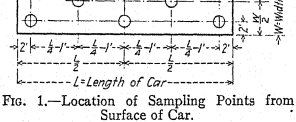


FIG. 1.—Location of Sampling Points from Surface of Car.

(*b*) In collecting the gross sample from a carload of mineral granules shipped in bags, a number of bags shall be selected at random, equivalent to the cube root of the total number of bags in the car. The gross sample shall consist of equal increments of not less than 2 lb. from each of the bags taken for sampling.

(*c*) Where the mineral granules are being loaded or unloaded by conveyor or chute the gross sample shall preferably be collected by taking equal increments from the stream at regular time intervals, with such frequency that the total weight of the gross sample will be not less than 16 lb. Increments shall be taken from the full width and thickness of the stream, preferably, if practicable, by inserting a suitable container into the stream.

TABLE I.—NOMINAL DIMENSIONS, PERMISSIBLE VARIATIONS, AND LIMITS FOR WOVEN WIRE CLOTH OF STANDARD SIEVES.

Size or Sieve Designation	Sieve Opening		Permissible Variation in Average Opening, per cent	Permissible Variation in Maximum Opening, per cent	Wire Diameter	
	mm.	in. (approx. equivalents)			mm.	in. (approx. equivalents)
3360 micron (No. 6).....	3.36	0.132	±3	+10	0.87 to 1.32	0.034 to 0.052
2380 micron (No. 8).....	2.38	0.0937	±3	+10	0.74 to 1.10	0.0291 to 0.0433
1680 micron (No. 12).....	1.68	0.0661	±3	+10	0.62 to 0.90	0.0244 to 0.0354
1190 micron (No. 16).....	1.19	0.0469	±3	+10	0.50 to 0.70	0.0197 to 0.0276
840 micron (No. 20).....	0.84	0.0331	±5	+15[a]	0.38 to 0.55	0.0150 to 0.0217
590 micron (No. 30).....	0.59	0.0232	±5	+15[a]	0.29 to 0.42	0.0114 to 0.0165
420 micron (No. 40).....	0.42	0.0165	±5	+25[a]	0.23 to 0.33	0.0091 to 0.0130
297 micron (No. 50).....	0.297	0.0117	±5	+25[a]	0.170 to 0.253	0.0067 to 0.0100
210 micron (No. 70).....	0.210	0.0083	±5	+25[a]	0.130 to 0.187	0.0051 to 0.0074
149 micron (No. 100).....	0.149	0.0059	±6	+40[a]	0.096 to 0.125	0.0038 to 0.0049

[a] Not more than 5 per cent of the openings shall exceed the nominal opening by more than one half of the permissible variation in maximum opening.

Reduction of Gross Sample

5. The gross sample shall be reduced by riffling or hand-quartering to not less than 500 g. (1.1 lb.).

Size of Test Sample

6. The entire sample obtained from reduction of the gross sample, shall be weighed with an accuracy of not less than 0.1 g., and taken for the sieve analysis.

Sieve Analysis

7. (*a*) The group of sieves selected from the series listed in Table I shall be assembled in consecutive order as to size of openings, with the sieve having the largest openings at the top and the

one with the smallest openings at the bottom, the assembly being completed by a solid collecting pan below the bottom sieve. The test sample, previously weighed (Section 6), shall be placed on the top sieve and this sieve closed with a solid cover. The sieve assembly shall then be securely fastened in a suitable mechanical sieve shaking device (Section 2 (*b*)).

(*b*) The sample shall be passed through the sieves of the series selected, by subjecting it to the action of the sieve shaker for a period of 15 min. Since granular mineral surfacing materials usually have been subjected to grading by screens in the process of manufacture and hence separate rapidly into their sieve fractions, an end point determination is not considered necessary.

(*c*) The portion of the sample retained on each of the sieves and on the pan shall be carefully removed and weighed with an accuracy of not less than 0.1 g.

Report

8. The results of the sieve analysis shall be reported to the nearest 0.1 per cent (omitting the results on those sieves on which less than 0.05 per cent of the total weight of the test sample was collected), as follows:

Retained on Sieve	Passing Sieve	Per Cent
3360-micron (No. 6)		.....
2380-micron (No. 8)	3360-micron (No. 6)	.....
1680-micron (No. 12)	2380-micron (No. 8)	.....
1190-micron (No. 16)	1680-micron (No. 12)	.....
840-micron (No. 20)	1190-micron (No. 16)	.....
590-micron (No. 30)	840-micron (No. 20)	.....
420-micron (No. 40)	590-micron (No. 30)	.....
297-micron (No. 50)	420-micron (No. 40)	.....
210-micron (No. 70)	297-micron (No. 50)	.....
149-micron (No. 100)	210-micron (No. 70)	.....
	149-micron (No. 100)	.....
Total		.....

The sum of the sieve weight percentage fractions, reported as above tabulated, shall be not less than 99.5 per cent, that is, the total loss and error in the sieve analysis shall not exceed 0.5 per cent.

Reproducibility of Results

9. Duplicate determinations by the same operator, using the same sieves, shall check within 1 per cent of the total weight of the sample, for the portion of the sample retained on each sieve and on the pan.

Standard Method of Test for

SIEVE ANALYSIS OF NONGRANULAR MINERAL SURFACING FOR ASPHALT ROOFING AND SHINGLES[1]

A.S.T.M. Designation: D 452 – 40

ADOPTED, 1940.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 452; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method is intended for the sieve analysis of nongranular mineral surfacing material, such as mica, talc and other powdered or flaky mineral particles, used on the weather surfaces of prepared asphalt roofing and on the non-weather-exposed surface of asphalt shingles.

Apparatus

2. (*a*) *Sieves.*—A set of consecutive sieves of the series listed in Table I, conforming to the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials,[3] shall be used. The group of sieves selected shall include those appropriate to the grading of the nongranular mineral to be subjected to sieve analysis. Sieves of either the coarser or finer openings, on which less than 0.05 per cent of the weight of the sample of the particular material would be found after sieving, need not be included in the set (Note). The wire cloth for these sieves shall be woven (not twilled) from brass, bronze, or other suitable wire, and shall be mounted without distortion in circular frames, 8 in. (20.32 cm.) in diameter and about 2 in. (5 cm.) between the top of the frame and the cloth.

NOTE.—For relatively coarse or flaky materials, such as coarse mica, sieves from 1190 to 210-micron (Nos. 16 to 70) inclusive, will usually be found suitable; for finer surfacing materials, such as fine mica or talc flour, sieves from 590 to 74-micron (Nos. 30 to 200) inclusive, will usually give a satisfactory sieve analysis.

(*b*) *Sieve Shaker.*—A mechanically operated sieve shaker, which imparts to the set of sieves a rotary motion and tapping action of uniform speed, shall be used. The number of taps per minute shall be between 140 and 160. The sieve shaker shall be fitted with a hard maple plug to receive the impact of the tapping device. The entire apparatus shall be rigidly mounted by bolting to

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] Prior to adoption as standard, this method was published as tentative from 1937 to 1940.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

a solid foundation, preferably of concrete.

(*c*) *Sample Splitter.*—A riffle sampler with $\frac{3}{8}$ or $\frac{1}{2}$-in. divisions, for reducing the gross sample to the quantity required for the sieve analysis, shall be used.

Unit for Sampling

3. Each carload of nongranular mineral surfacing shall be considered the unit for sampling.

Collection of Gross Sample

4. (*a*) In collecting the gross sample from a carload of nongranular mineral surfacing shipped in bags, a number of bags shall be selected at random equivalent to the cube root of the total number of bags in the car. The gross sample shall consist of equal increments of not less than 0.5 lb. from each of the bags taken for sampling.

(*b*) In collecting the gross sample from a carload of nongranular mineral surfacing material shipped in bulk, at least eight equal increments of not less than 0.5 lb. each shall be taken from well distributed points in the mass, using a sampling tube not less than 1 in. in diameter. A conical excavation about 1 ft. in depth shall be made at each sampling point and the sampling tube inserted into the mass at the bottom of the excavation.

Reduction of Gross Sample

5. The gross sample shall be reduced by riffling or hand-quartering to not less than 100 g. (0.22 lb.).

Size of Test Sample

6. The entire sample obtained from reduction of the gross sample, shall be dried at least 2 hr. in an oven at 220 F., weighed with an accuracy of not less than 0.1 g., and taken for the sieve analysis.

TABLE I.—NOMINAL DIMENSIONS, PERMISSIBLE VARIATIONS, AND LIMITS FOR WOVEN WIRE CLOTH OF STANDARD SIEVES.

Size or Sieve Designation	Sieve Opening		Permissible Variation in Average Opening, per cent	Permissible Variation in Maximum Opening, per cent	Wire Diameter	
	mm.	in. (approx. equivalents)			mm.	in. (approx equivalents)
1680 micron (No. 12).....	1.68	0.0661	±3	+10	0.62 to 0.90	0.0244 to 0.0354
1190 micron (No. 16).....	1.19	0.0469	±3	+10	0.50 to 0.70	0.0197 to 0.0276
840 micron (No. 20).....	0.84	0.0331	±5	+15[a]	0.38 to 0.55	0.0150 to 0.0217
590 micron (No. 30).....	0.59	0.0232	±5	+15[a]	0.29 to 0.42	0.0114 to 0.0165
420 micron (No. 40).....	0 42	0.0165	±5	+25[a]	0.23 to 0.33	0.0091 to 0.0130
297 micron (No. 50).....	0.297	0.0117	±5	+25[a]	0.170 to 0.253	0.0067 to 0.0100
210 micron (No. 70).....	0.210	0.0083	±5	+25[a]	0.130 to 0.187	0.0051 to 0.0074
149 micron (No. 100).....	0.149	0.0059	±6	+40[a]	0.096 to 0.125	0.0038 to 0.0049
74 micron (No. 200).....	0.074	0.0029	±7	+60[a]	0.045 to 0.061	0.0018 to 0.0024

[a] Not more than 5 per cent of the openings shall exceed the nominal opening by more than one half of the permissible variation in maximum opening.

Sieve Analysis

7. (*a*) The group of sieves selected from the series listed in Table I shall be assembled in consecutive order as to size of openings, with the sieve having the largest openings at the top and the one with the smallest openings at the bottom, the assembly being completed by a solid collecting pan below the bottom sieve. The test sample, previously weighed (Section 6), shall be placed on the top sieve and this sieve closed with a solid cover. The sieve assembly shall then be securely fastened in a suitable mechanical sieve shaking device (Section 2 (*b*)).

(*b*) The sample shall be passed through the sieves of the series selected, by subjecting it to the action of the sieve shaker for a period of 20 min. At the end of this period the collecting pan, containing the portion of the material passing the finest sieve of the group selected, shall be removed from the sieve assembly and the contents weighed with an accuracy of not less than 0.1 g. The collecting pan shall then be re-assembled with the sieves, as before, and the shaking continued for an additional 10 min. At the end of this additional shaking period the collecting pan shall be removed and the contents weighed. If the additional material passing the finest sieve during this second shaking period does not exceed 0.5 per cent of the total weight of the sample, the sieve analysis shall be considered complete. If it does exceed 0.5 per cent, the collecting pan and sieves shall be re-assembled and shaken for successive additional 10-min. periods, weighing the material collected in the pan after each period of shaking, until the amount passing the finest sieve in a 10-min. shaking period is less than 0.5 per cent of the weight of the sample.

(*c*) The portion of the sample retained on each of the sieves and on the pan shall be carefully removed and weighed with an accuracy of not less than 0.1 g.

Report

8. The results of the sieve analysis shall be reported to the nearest 0.1 per cent (omitting the results on those sieves on which less than 0.05 per cent of the total weight of the test sample was collected), as follows:

Retained on Sieve	Passing Sieve	Per Cent
1680-micron (No. 12)	.....	.....
1190-micron (No. 16)	1680-micron (No. 12)	.....
840-micron (No. 20)	1190-micron (No. 16)	.....
590-micron (No. 30)	840-micron (No. 20)	.....
420-micron (No. 40)	590-micron (No. 30)	.....
297-micron (No. 50)	420-micron (No. 40)	.....
210-micron (No. 70)	297-micron (No. 50)	.....
149-micron (No. 100)	210-micron (No. 70)	.....
74-micron (No. 200)	149-micron (No. 100)	.....
	74-micron (No. 200)	.....
Total	.....	.....

The sum of the sieve weight percentage fractions, reported as above tabulated, shall be not less than 98.5 per cent, that is, the total loss and error in the sieve analysis shall not exceed 1.5 per cent.

Reproducibility of Results

9. Duplicate determinations by the same operator, using the same sieves, shall check within 1 per cent of the total weight of the sample, for the portion of the sample retained on each sieve and on the pan.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: 06-1939

Standard Specifications for
ROUND TIMBER PILES[1]



A.S.T.M. Designation: D 25 - 37

ADOPTED, 1937.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 25; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover round timber piles to be used untreated, or treated by standard preservatives.

NOTE.—Where sawed timber is used as piling, such as heavy sheet piles, appropriate specifications should be selected from the Standard Specifications for Structural Wood Joist and Plank, Beams and Stringers, and Posts and Timbers (A.S.T.M. Designation: D 245) of the American Society for Testing Materials.[3]

Kinds of Wood

2. (*a*) The purchaser shall specify the kind or kinds of wood he desires, and shall designate the kinds he desires for preservative treatment.

NOTE.—Commonly used species are cedars, chestnut, cypress, Douglas fir, larch, oaks, pines, spruces, and tamarack.

(*b*) Piles of different kinds of wood shall be delivered in separate lots.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-7 on Timber.

[2] Prior to their present adoption as standard, these specifications were published as tentative from 1915 to 1920. They were adopted in 1920, published as standard from 1920 to 1930, but withdrawn and republished as tentative from 1930 to 1937, being revised in 1934 and 1935.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Use Classification

3. Timber piles are classified in these specifications under three general divisions according to the use intended, as follows:

(*a*) *Class A.*—Piles suitable for use in heavy railway bridges and trestles. The minimum diameter of butt assumes the use of load-bearing timber caps 14 in. in width.

(*b*) *Class B.*—Piles suitable for use in docks, wharves, highway work, and general construction. The minimum diameter of butt assumes the use of load-bearing timber caps 12 in. in width. When timber caps are not used, as in the case of piles under masonry foundations, the sizes given for Class C piles are recommended.

(*c*) *Class C.*—Piles suitable for use in foundations which will always be completely submerged, for coffer-dams, falsework, and sundry temporary work.

Class A and Class B Piles

General Quality

4. Except as hereinafter provided, Class A and Class B piles shall be free

from any defects which may impair their strength or durability as piling, such as decay, red heart, splits in piles to be treated, or splits longer than the measured butt diameter of piles not to be treated, twist of grain exceeding one-half of the circumference in any 20 ft. of length, unsound knots, numerous knots or holes, or shake more than one-third of the diameter of the pile. Piles which have been scored for turpentine shall be accepted, provided such scar does not exceed 36 in., and provided the scoring is of recent date showing the scar to be entirely sound and free from insect damage.

Knots

5. Sound knots will be permitted in Class A and Class B piles, provided they are not in clusters. The diameter of a sound knot shall not be greater than one-third of the minimum diameter of the pile at the section where it occurs, and shall not exceed 4 in. for piles 50 ft. and under in length. For piles over 50 ft. in length, knots between the section at mid-length and the butt shall conform to the limitation prescribed for piling under 50 ft. Between mid-length and the tip, single knots up to 5 in. in diameter will be permitted, provided they do not exceed one-half the minimum diameter of the pile at the section where they occur. The diameter of a knot shall be measured at right angles to the length of the pile.

General Requirements

6. (*a*) *Sound Timber.*—Class A and Class B piles shall be cut from sound, live trees, except that fire-killed, blight-killed, or wind-felled timber may be used if not attacked by decay or insects. Piles shall be cut above the ground swell.

(*b*) *Tip.*—The tip shall be sound.

(*c*) *Butt End.*—The butt end shall be sound except in cedar piles, which may have a pipe or stump rot hole not more than 1½ in. in diameter.

(*d*) *Taper.*—Piles shall have a gradual taper from the point of butt measurement to the tip.

(*e*) *Surface Finish.*—All knots and limbs shall be trimmed or smoothly cut flush with the surface of the pile. The butt and tip shall be sawed square with the axis of the pile, or the tip may be tapered to a point not less than 4 in. in diameter if directed by the engineer in charge.

Sapwood

7. (*a*) *Piles for Use Untreated.*—Piles for use without preservative treatment shall have as little sapwood as possible, and when used in exposed work, the diameter of the heartwood shall not be less than eight-tenths of the actual diameter of the pile at the butt.

(*b*) *Piles for Treatment.*—Piles for use with preservative treatment shall have no sapwood restrictions, but preferably shall contain as much sapwood as possible. In southern pine the sapwood thickness shall not be less than 1½ in. and in Douglas fir and larch not less than 1 in. on the butt end.

Peeled Piles

8. (*a*) Piles shall be peeled of bark, including the inner skin, soon after cutting so that the piles are smooth and clean. Care shall be taken to remove as little sapwood as possible while peeling the bark. The sapwood shall not be injured by unnecessary axe cuts. These piles shall be designated as piles for treatment.

(*b*) No pile shall be considered as thoroughly peeled unless all of the rough bark and at least 80 per cent of the inner bark which remains on the pile shall have been removed. In no case shall any piece of inner bark be over ¾ in. in width or over 8 in. in length, and

there shall be 1 in. of clean wood surface between any two strips of inner bark.

Diameter

9. (*a*) It is recommended that the diameters of piles measured under the bark shall conform to the requirements shown in Table I, subject to a permissible

TABLE I.—DIAMETERS OF THREE CLASSES OF PILES.

Length, ft.	Class A and Class B Piles (These differ in size only.)						Class C Piles[a] (These differ from Classes A and B in size and quality.)		
	Class A			Class B					
	Diameter 3 ft. from Butt, in.		Diameter of Tip, in.	Diameter 3 ft. from Butt, in.		Diameter of Tip, in.	Diameter 3 ft. from Butt, in.		Diameter of Tip, in.
	Minimum	Maximum	Minimum	Minimum	Maximum	Minimum	Minimum	Maximum	Minimum
DOUGLAS FIR AND SOUTHERN PINE PILES[b]									
Under 40.....	14	18	10	12	20	8	12	20	8
40 to 50, incl..	14	18	9	12	20	7	12	20	6
51 to 70, incl..	14	18	8	13	20	7	12	20	6
71 to 90, incl..	14	20	7	13	20	6	12	20	6
Over 90.....	14	20	6	13	20	5	12	20	5
BLACK OAK, CHESTNUT, CYPRESS, PIN OAK, POST OR BURR OAK, RED OAK, WHITE OAK, OR WILLOW OAK PILES									
Under 30.....	14	18	10	12	18	8	12	20	8
30 to 40, incl..	14	18	9	13	20	8	12	20	8
Over 40.....	14	18	8	13	20	7	12	20	6
CEDAR PILES									
Under 30.....	14	22	10	12	22	8	12	22	8
30 to 40, incl..	14	22	9	13	22	8	12	22	8
Over 40.....	14	22	8	13	22	7	12	22	7

[a] In Class C piles, a minimum diameter (at cut-off) of 10 in. may be specified for lengths of 30 ft. and under.
[b] Where larch, lodgepole or Norway pine, spruce, or tamarack piles are specified, their dimensions shall correspond to the requirements shown for Douglas fir and southern pine.

variation of minus $\frac{1}{2}$ in. in any diameter in not more than 25 per cent of the piles of that diameter.

(*b*) The diameter of a pile in cases where the tree is not exactly round shall be determined either by measuring the circumference and dividing the number of inches by 3.14, or by taking the average of the maximum and minimum diameters at the location specified.

Length

10. All piles shall be furnished cut to any of the following lengths, as specified: 16 to 40 ft. in multiples of 2 ft., and over 40 ft. in multiples of 5 ft. A variation of 6 in. in length shall be allowable, but the average length in any shipment shall be equal to, or greater than, the specified lengths. The length of each pile shall be legibly marked on the butt end with white or black paint.

Straightness

11. Piles shall be free from short or reversed bends, and free from crooks greater than one-half of the diameter of the pile at the middle of the bend. In short bends, the distance from the center of the pile to a line stretched from the center of the pile above the bend to the center of the pile below the bend shall not exceed 4 per cent of the length of the bend, or $2\frac{1}{2}$ in. A line drawn from the center of the butt end to the center of the tip shall lie within the body of the pile.

Class C Piles

General Quality

12. Class C piles shall be of sound, live timber that will stand driving, and need not be peeled if they are to be used without preservative treatment. They shall be free from decay and other imperfections such as bad knots and shakes which will materially affect their strength. Piles which have been scored for turpentine shall be accepted, provided such scar does not exceed 36 in., and provided the scoring is of recent date showing the scar to be entirely sound and free from insect damage.

General Requirements

13. (*a*) *Tip.*—The tip shall be sound.

(*b*) *Taper.*—Piles shall have a gradual taper from the point of butt measurement to the tip.

(*c*) *Surface Finish.*—All knots and limbs shall be trimmed or smoothly cut flush with the surface of the pile. The butt and tip shall be sawed square with the axis of the pile, or the tip may be tapered to a point not less than 4 in. in diameter if directed by the engineer in charge.

Sapwood

14. Sapwood requirements shall be identical with those for Class A and Class B piles as specified in Section 7.

Peeled Piles

15. The requirements for peeled piles shall be identical with those for Class A and Class B piles as specified in Section 8.

Diameter

16. The methods of measurement of diameter and the permissible variations in diameter shall be identical with those for Class A and Class B piles as specified in Section 9. The recommended sizes are given in Table I.

Length

17. The requirements for length shall be identical with those for Class A and Class B piles as specified in Section 10.

Straightness

18. The requirements relating to straightness shall be identical with those for Class A and Class B piles as specified in Section 11.

Standard Specifications for

WOODEN PAVING BLOCKS FOR EXPOSED PAVEMENTS[1]

A.S.T.M. Designation: D 52 – 20

ADOPTED, 1920.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 52; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover wooden paving blocks for pavements exposed to alternate wet and dry conditions, as distinguished from pavements which are used under cover and protected from atmospheric influences.

Kind of Timber

2. The wood, which shall be treated, shall be southern pine, Douglas fir, tamarack, Norway pine, hemlock, or black gum. Only one kind of wood shall be used in any one contract. The blocks shall be sound; shall be well manufactured, square-butted, and square-edged; and shall be free from unsound, loose, or hollow knots, knot holes, worm holes, and other defects such as shakes, checks, etc. that would be detrimental to the blocks.

Quality of Timber

3. (*a*) *Number of Annual Rings.*—The number of annual rings in the 1 in. which begins 2 in. from the pith of the block shall not be less than six, measured radially; provided, however, that blocks containing between five and six rings in this inch shall be accepted if they contain 33⅓ per cent or more summerwood. In case the block does not contain the pith, the 1 in. to be used shall begin 1 in. away from the ring which is nearest to the heart of the block.

(*b*) *Heartwood.*—The blocks in each charge shall contain an average of at least 70 per cent of heartwood. No one block shall be accepted that contains less than 50 per cent of heartwood.

Size of Blocks

4. The blocks shall be from 5 to 10 in. in length, but should preferably average two times the depth; they shall be .... in. in depth.[3] They may be from 3 to 4 in. in width, but in any one city square all of them shall be of uniform width. Permissible variations from the specified dimensions shall not

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-7 on Timber.

[2] Prior to adoption as standard, these specifications were published as tentative from 1918 to 1920, being revised in 1920. Editorially revised and rearranged in 1939.

[3] It is recommended that blocks 4 in. in depth be used for streets with very heavy traffic, and blocks 3½ in. in depth for streets with moderate traffic. For streets with light traffic, blocks 3 in. in depth may be used, but where 3-in. blocks are used no block shall be longer than 8 in.

exceed $\frac{1}{16}$ in. in depth and $\frac{1}{8}$ in. in width, except that in all cases the width shall be greater or less than the depth by at least $\frac{1}{4}$ in.

Preservative

5. The preservative used shall be coal tar paving oil or distillate oil, as specified by the purchaser.

Preservative Treatment

6. (*a*) The timber may be either air-seasoned or green, but should preferably be treated within three months from the time it is sawed. Green timber and seasoned timber shall not be treated together in the same charge. The blocks shall be treated in an airtight cylinder with the preservative specified by the purchaser. In all cases, whether thoroughly air-seasoned or green, they shall be first subjected to live steam at a temperature between 220 and 240 F.[4] for not less than 2 nor more than 4 hr. at the discretion of the treating plant operator, after which they shall be subjected to a vacuum of not less than 22 in. maintained for at least 1 hr. While the vacuum is still on, the preservative oil, heated to a temperature of between 180 and 220 F., shall be run in until the cylinder is completely filled, care being taken that no air is admitted. Pressure shall then be gradually applied not to exceed 50 psi. at the end of the first hour nor 100 psi. at the end of the second hour, and then maintained at not less than 100 psi. nor more than 150 psi. until the wood has absorbed the required amount of oil.[5]

(*b*) After this a supplemental vacuum, in which the maximum intensity reached is at least 20 in. and the time the vacuum is applied not less than 30 min., shall be applied. If desired, this vacuum may be followed by a short steaming period.

Penetration of Preservative

7. In any charge, blocks shall contain at least 16 lb. of water-free oil per cubic foot of wood at the completion of the treatment. The blocks after treatment shall show satisfactory penetration of the preservative, and in all cases the oil shall be diffused throughout the sapwood. To determine this, at least 25 blocks shall be selected from various parts of each charge and sawed in half perpendicular to the fibers through the center, and if more than one of these blocks show untreated sapwood, the charge shall be re-treated. After re-treating, the charge shall be again subjected to a similar inspection.

Handling Blocks After Treatment

8. Blocks shall preferably be laid in the street as soon as possible after being treated. If they cannot be laid immediately, provision shall be made to prevent them from drying out by stacking in close piles and covering them, and, if possible, by sprinkling them thoroughly at intervals. In any case, when they are not laid as soon as they are received at the site of the work, they shall be well sprinkled under the direction of the purchaser about two days before being laid. It is important to have the wood sufficiently wet to be swelled to its maximum size before it is laid.

Inspection at Plant

9. All material prescribed in these specifications and processes used in the manufacture of the blocks therefrom shall be subject to inspection, acceptance, or rejection at the plant of the manufacturer, which shall be equipped with all the necessary gages, appliances,

[4] In no case shall a steam pressure of 20 psi. be exceeded.

[5] This treatment is recommended for southern pine only. It is probably also suited to Norway pine, hemlock, black gum, and tamarack, but not to Douglas fir. Further recommendations on the treatment of these species are reserved for the future.

and facilities to enable the inspector to satisfy himself that the requirements of the specifications are being fulfilled.

Inspection at Site of Work

10. The purchaser shall have the further right to inspect the blocks after delivery at the site of the work, for the purpose of rejecting any blocks that do not conform to these specifications; except that the inspections at the plant shall be final with respect to the kind of wood, number of rings per inch, oil, and treatment.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: O7-1939

Standard Specifications for

STRUCTURAL WOOD JOIST AND PLANK, BEAMS AND STRINGERS, AND POSTS AND TIMBERS[1]

A.S.T.M. Designation: D 245 – 37

ADOPTED, 1937.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 245; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

These specifications were prepared jointly by the American Railway Engineering Association and the American Society for Testing Materials

GENERAL REQUIREMENTS

Applying to All Stress-Grades and Items

Manufacture

1. All material shall be well manufactured.

Quality of Wood

2. No piece of exceptionally light weight shall be permitted.

Decay

3. Only pieces consisting of sound wood free from any form of decay shall be acceptable, unless otherwise specified.

Slope of Grain

4. Slope of grain shall be measured over a distance sufficiently great to determine the general slope disregarding slight local deviations. Within the middle half of length of piece, slope of grain shall not be steeper than specified.

Holes

5. Knot holes and holes from causes other than knots shall be measured and limited as provided for knots.

Cluster Knots

6. Cluster knots and knots in groups shall not be permitted.

Knots in Joist and Plank

7. (*a*) *Knots on Narrow Faces.*—Size of knot on a narrow face shall be taken as the width between lines enclosing the knot and parallel to the edges of the piece. The only knots measured on narrow faces, except spike knots which cross the corners of side-

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-7 on Timber.

[2] Prior to their present adoption as standard, these specifications were published as tentative from 1926 to 1927, being revised in 1927. They were adopted in 1927, revised in 1929, 1930, and 1933, but withdrawn and republished as tentative from 1936 to 1937. Editorially revised and rearranged in 1939.

cut pieces, shall be those that do not show on wide faces.

(*b*) *Knots on Wide Faces.*—Size of knot on a wide face shall be the average of its largest and smallest diameters. A spike knot which crosses a corner of a side-cut piece and contains the intersection of the adjacent faces, or which extends entirely across a face of a piece, shall be measured only on its end or ends, between lines parallel to the edges of the piece.

(*c*) *Permissible Increase in Knot Sizes.*—Size of knots on narrow faces and at edges of wide faces may increase proportionately from the size permitted in the middle third of the length to twice that size at ends of the piece. Size of knots on wide faces may increase proportionately from the size permitted at the edge to the size permitted along the center line.

(*d*) *Sum of Knots.*—Sum of sizes of all knots within the middle half of the length of any face, measured as specified in Paragraph (*a*) or (*b*) for the face under consideration, shall not exceed four and one-half times the size of the largest knot allowed on that face.

Knots in Beams and Stringers

8. (*a*) *Knots on Narrow Faces.*—Size of knot on a narrow face shall be taken as the width between the lines enclosing the knot and parallel to the edges of the piece except that when a knot on a narrow face extends into the adjacent one-fourth of the width of a wide face its least dimension shall be taken as its size.

(*b*) *Knots on Wide Faces.*—Size of knot on a wide face shall be its smallest diameter. Knots at the edges of wide faces shall be limited to the same sizes as on narrow faces but are measured as specified in this paragraph.

(*c*) *Permissible Increase in Knot Size.*—Size of knots on narrow faces and at the edges of wide faces may increase proportionately from the size permitted in the middle third of the length to twice that size at the ends of the piece, except that no knot shall exceed the size permitted along the center line of a wide face. Size of knots on wide faces may increase proportionately from the size permitted at the edge to the size permitted along the center line.

(*d*) *Sum of Knots.*—Sum of sizes of all knots within the middle half of the length of any face, measured as specified in Paragraph (*a*) or (*b*) for the face under consideration, shall not exceed four times the size of the largest knot allowed on that face.

Knots in Posts and Timbers

9. (*a*) *Knots on Either Face.*—Size of a knot on either face shall be half the sum of its largest and smallest diameters. A spike knot which crosses a corner of a side-cut piece and contains the intersection of the adjacent faces, or which extends entirely across a face of a piece, shall be measured only on its end or ends, between lines parallel to the edges of the piece.

(*b*) *Sum of Knots.*—Sum of sizes of all knots in any 6 in. of the length of piece, measured as specified in Paragraph (*a*), shall not be permitted to exceed twice the maximum permissible size of knot. Two knots of the maximum permissible size shall not be permitted in the same 6 in. of length on any face.

Shakes, Checks, Splits in Joist and Plank and Beams and Stringers

10. (*a*) Shakes, checks, and splits, when permitted in the grade specified later in these specifications, shall be measured at the ends of the piece. Only those within the middle half of the height shall be considered. (The height equals the width of the wide face.) Size of shake is the distance between the lines enclosing the shake and parallel to the wide faces of the piece. Per-

missible size is determined by the width of the narrow face of the piece.

(*b*) Checks and splits shall be measured and limited in the same way as shakes. The following limitations apply to both ends but only within the middle half of the height of the piece and within three times the height from the end. (The height equals the width of the wide face.) Size of checks within this portion of the piece shall be taken as their estimated area, along the horizontal section showing maximum area, divided by three times the height of the piece (Note). When checks on two parallel faces are opposite or approximately so, the sum of their sizes shall be taken. The sum of sizes of shakes, checks, and/or splits shall not exceed the permissible size of shake.

(*c*) Checks extending entirely across the end within the middle half of the height shall not extend into the piece at the center of width of end a distance greater than the size of the allowable shake.

Shakes, Checks, Splits in Posts and Timbers

11. (*a*) Shakes, checks, and splits, when permitted in the grade specified later in these specifications, shall be measured at the ends of the piece. Size of a shake is the distance between the lines enclosing the shake and parallel to a pair of opposite faces.

(*b*) Checks and splits shall be measured and limited in the same way as shakes. Size of checks within three times the width of the piece from either end shall be taken as their estimated area, along the longitudinal section showing maximum area, divided by three times the width of the piece (Note). Sum of sizes of shakes, checks, and/or splits shall not exceed the permissible size of shake.

(*c*) Checks extending entirely across the end shall not extend into the piece at the center of width of end, a distance greater than the size of allowable shake.

NOTE.—A practical method of inspection in the field to determine the size of checks and splits under the above specification is as follows:

The size of checks within the specified portion of the piece shall be taken as the sum of seven depth measurements, one on the end and three on each side, divided by three. Each measurement shall represent the greatest depth of *any* check within the center half of the height; the measurement on the end shall be taken at the center of the width, and the three measurements on each side shall be taken at distances of one, two, and three times the height of the piece from the end in joist, plank, beams and stringers, and the width of the piece from the end in posts and timbers. Each measurement shall be determined by the penetration into the piece of a probe $\frac{1}{64}$ in. in thickness and $\frac{1}{2}$ in. in width.

OPTIONAL AND ADDITIONAL REQUIREMENTS

Applying to Stress-Grades when Heartwood or Wane, and When Density and Close Grain Are Required

Heartwood Requirements

12. (*a*) When heartwood material is desired it shall be specified in terms of the amount or percentage of heartwood on the girth, or on each face, side, or edge, measured at the point where the greatest amount of sapwood occurs.

(*b*) For all timber to be pressure treated there shall be no heartwood requirement and the amount of sapwood shall not be limited.

Wane

13. When wane is not desired, the specifications should state "Square Edge."

Density (when required) of Dense Douglas Fir

14. (*a*) Dense Douglas Fir shall average on either one end or the other of each piece not less than six annual rings

per inch, and, in addition, one-third or more summerwood (the darker, harder portion of the annual ring) measured over 3 in. on a line at right angles to the annual rings, located as described below. The contrast in color between summerwood and springwood shall be distinct.

(*b*) Coarse-grained material excluded by this rule shall be accepted as dense if averaging one-half or more summerwood.

(*c*) In boxed-heart pieces the line shall run from the pith to the corner farthest from the pith, except that when the line is not representative it shall be shifted sufficiently to present a fair average, but the distance from the pith to the beginning of the 3-in. portion of the line shall not be changed. When the least dimension is 6 in. or less, the 3-in. portion of the line shall begin at a distance of 1 in. from the pith. When the least dimension is more than 6 in., the 3-in. portion of the line shall begin at a distance from the pith equal to one-fourth the least dimension of the piece.

(*d*) In side-cut pieces (pith not present) the center of the 3-in. portion of the line shall be at the center of the end of the piece; if a 3-in. portion of the line cannot be obtained, the measurement shall be made over as much of a 3-in. portion as is available.

(*e*) In case of disagreement, two radial lines shall be chosen and the summerwood and number of rings shall be taken as the average on these lines.

Density (when required) of Dense Longleaf or Shortleaf Yellow Pine

15. (*a*) Dense longleaf or shortleaf yellow pine shall average on either one end or the other of each piece not less than six annual rings per inch, and, in addition, one-third or more summerwood (the darker, harder portion of the annual ring), measured over the third, fourth, and fifth inches of a radial line from the pith. The contrast in color between summerwood and springwood shall be sharp and the summerwood shall be dark in color, except in pieces having considerably above the minimum requirement for summerwood.

(*b*) Coarse-grained material excluded by this rule shall be accepted as dense if averaging one-half or more summerwood.

(*c*) The radial line shall be representative of the average growth of the cross-section. In case of disagreement, two radial lines shall be chosen, and the number of rings per inch and percentage of summerwood shall be taken as the average determined on these lines.

(*d*) In boxed-heart pieces the measurement shall be made over the third, fourth, and fifth inches from the pith along the radial line.

(*e*) In material containing the pith, but not a 5-in. radial line, which is less than 2 by 8 in. in section or less than 8 in. in width, that does not show over 16 sq. in. on the cross-section, the inspection shall apply to the second inch from the pith. In larger material that does not show a 5-in. radial line, the inspection shall apply to the three inches farthest from the pith.

(*f*) In cases where timbers do not contain the pith and it is impossible to locate it with any degree of accuracy, the same inspection shall be made over 3 in. on an approximate radial line beginning at the edge nearest the pith in timbers over 3 in. in thickness and on the second inch nearest the pith in timbers 3 in. or less in thickness.

Close Grain (when required) of Close-Grained Douglas Fir, or Redwood

16. (*a*) Close-grained Douglas fir shall average on either one end or the other of each piece not less than six nor more than twenty annual rings per inch; close-grained redwood shall average on

either one end or the other of each piece not less than ten nor more than thirty-five annual rings per inch. The rings shall be measured over 3 in. on a line at right angles to the annual rings, located as described below.

(*b*) In boxed-heart pieces the line shall run from the pith to the corner farthest from the pith except that when the line is not representative it shall be shifted sufficiently to present a fair average, but the distance from the pith to the beginning of the 3-in. portion of the line shall not be changed. When the least dimension is 6 in. or less, the 3-in. portion of the line shall begin at a distance of 1 in. from the pith. When the least dimension is more than 6 in., the 3-in. portion of the line shall begin at a distance from the pith equal to one-fourth the least dimension of the piece.

(*c*) In side-cut pieces (pith not present) the center of the 3-in. portion of the line shall be at the center of the end of the piece. If a 3-in. portion of the line cannot be obtained, the measurement shall be made over as much of a 3-in. portion as is available.

(*d*) In case of disagreement, two radial lines shall be chosen and the number of rings shall be taken as the average on these lines.

(*e*) Pieces of Douglas fir averaging five rings, or more than twenty, shall be accepted if they contain one-third or more summerwood.

STRESS-GRADES

I. Joist and Plank

= Extreme fiber stress in bending in pounds per square inch for continuously dry locations.

1800 lb. *f* Structural Joist and Plank

DENSE DOUGLAS FIR (COAST REGION)
DENSE DOUGLAS FIR (INLAND)
DENSE LONGLEAF SOUTHERN PINE
DENSE SHORTLEAF SOUTHERN PINE

Slope of Grain.—1 in 12.

Knots.—Maximum permissible size in inches:

Nominal Width of Face, in.	On Narrow Face, or at Edge of Wide Face, Middle Third of Length	At Center Line of Wide Face
2.....	$\frac{5}{6}$ in. On	.....
3.....	1 in. Narrow	.....
4.....	$1\frac{1}{4}$ in. Face	.....
4.....	$\frac{3}{4}$ in.	$1\frac{1}{4}$ in.
6.....	1 in. At	$1\frac{7}{8}$ in.
8.....	$1\frac{3}{8}$ in. Edge	$2\frac{1}{2}$ in.
10.....	$1\frac{3}{4}$ in. of	$3\frac{1}{2}$ in.
12.....	$2\frac{1}{8}$ in. Wide	$3\frac{7}{8}$ in.
14.....	$2\frac{3}{8}$ in. Face	$4\frac{3}{8}$ in.
16.....	$2\frac{3}{4}$ in.	$4\frac{3}{4}$ in.

	Width of Narrow Face	
	When Green	When Seasoned
Shakes, Checks, Splits:		
120 lb. Shear Grade (Fir)	$\frac{1}{6}$	$\frac{1}{4}$
120 lb. Shear Grade (Pine)	$\frac{1}{10}$	$\frac{3}{8}$
Wane.....	$\frac{1}{6}$ Width of any face	

1600 lb. *f* Structural Joist and Plank

CLOSE-GRAINED DOUGLAS FIR (COAST REGION)

Slope of Grain.—1 in 12.

Knots.—Maximum permissible size in inches:

NOMINAL WIDTH OF FACE, IN.	ON NARROW FACE, OR AT EDGE OF WIDE FACE, MIDDLE THIRD OF LENGTH	AT CENTER LINE OF WIDE FACE
2	$\frac{5}{8}$ in. On	.....
3	1 in. Narrow	.....
4	$1\frac{1}{4}$ in. Face	.....
4	$\frac{3}{4}$ in.	$1\frac{1}{4}$ in.
6	1 in. At	$1\frac{7}{8}$ in.
8	$1\frac{3}{8}$ in. Edge	$2\frac{1}{2}$ in.
10	$1\frac{3}{4}$ in. of	$3\frac{1}{8}$ in.
12	$2\frac{1}{8}$ in. Wide	$3\frac{7}{8}$ in.
14	$2\frac{1}{4}$ in. Face	$4\frac{1}{2}$ in.
16	$2\frac{1}{2}$ in.	$4\frac{3}{2}$ in.

	WIDTH OF NARROW FACE	
	When Green	When Seasoned
Shakes, Checks, Splits:		
100 lb. Shear Grade	$\frac{1}{6}$	$\frac{1}{4}$
Wane	$\frac{1}{6}$ Width of any face	

DENSE LONGLEAF SOUTHERN PINE
DENSE SHORTLEAF SOUTHERN PINE

Slope of Grain.—1 in 12.

Knots.—Maximum permissible size in inches:

NOMINAL WIDTH OF FACE, IN.	ON NARROW FACE, OR AT EDGE OF WIDE FACE, MIDDLE THIRD OF LENGTH	AT CENTER LINE OF WIDE FACE
2	$\frac{3}{4}$ in. On	.....
3	$1\frac{1}{8}$ in. Narrow	.....
4	$1\frac{1}{2}$ in. Face	.....
4	$\frac{7}{8}$ in.	$1\frac{1}{2}$ in.
6	$1\frac{1}{2}$ in. At	$2\frac{1}{4}$ in.
8	$1\frac{7}{8}$ in. Edge	3 in.
10	$2\frac{3}{8}$ in. of	$3\frac{3}{4}$ in.
12	$2\frac{3}{4}$ in. Wide	$4\frac{1}{2}$ in.
14	$2\frac{7}{8}$ in. Face	$4\frac{3}{4}$ in.
16	$2\frac{7}{8}$ in.	$5\frac{1}{4}$ in.

	WIDTH OF NARROW FACE	
	When Green	When Seasoned
Shakes, Checks, Splits:		
120 lb. Shear Grade	$\frac{3}{16}$	$\frac{3}{8}$
Wane	$\frac{1}{8}$ Width of any face	

CLOSE-GRAINED REDWOOD

Slope of Grain.—1 in 16.

Knots.—Maximum permissible size in inches:

NOMINAL WIDTH OF FACE, IN.	ON NARROW FACE, OR AT EDGE OF WIDE FACE, MIDDLE THIRD OF LENGTH	AT CENTER LINE OF WIDE FACE
2	$\frac{3}{8}$ in. On	.....
3	$\frac{5}{8}$ in. Narrow	.....
4	$\frac{7}{8}$ in. Face	.....
4	$\frac{3}{8}$ in.	$\frac{7}{8}$ in.
6	$\frac{5}{8}$ in. At	$1\frac{1}{4}$ in.
8	$\frac{7}{8}$ in. Edge	$1\frac{3}{4}$ in.
10	$1\frac{1}{8}$ in. of	$2\frac{1}{8}$ in.
12	$1\frac{1}{4}$ in. Wide	$2\frac{1}{2}$ in.
14	$1\frac{3}{8}$ in. Face	$2\frac{3}{4}$ in.
16	$1\frac{1}{2}$ in.	3 in.

	WIDTH OF NARROW FACE	
	When Green	When Seasoned
Shakes, Checks, Splits:		
80 lb. Shear Grade	$\frac{1}{8}$	$\frac{2}{8}$
Wane	$\frac{1}{8}$ Width of any face	

1400 lb. *f* Structural Joist and Plank

TIDEWATER RED CYPRESS

Slope of Grain.—1 in 14.

Knots and Peck.—Maximum permissible size in inches:

NOMINAL WIDTH OF FACE, IN.	ON NARROW FACE, OR AT EDGE OF WIDE FACE, MIDDLE THIRD OF LENGTH	AT CENTER LINE OF WIDE FACE
2	$\frac{5}{8}$ in.	On	.....
3	$\frac{5}{8}$ in.	Narrow	.....
4	$1\frac{1}{8}$ in.	Face	.....
4	$\frac{5}{8}$ in.		$1\frac{1}{8}$ in.
6	1 in.	At	$1\frac{1}{4}$ in.
8	$1\frac{1}{2}$ in.	Edge	$2\frac{3}{8}$ in.
10	$1\frac{5}{8}$ in.	of	$2\frac{7}{8}$ in.
12	$1\frac{5}{8}$ in.	Wide	$3\frac{1}{2}$ in.
14	2 in.	Face	$3\frac{7}{8}$ in.
16	$2\frac{1}{4}$ in.		$4\frac{1}{8}$ in.

	WIDTH OF NARROW FACE When Green	When Seasoned
Shakes, Checks, Splits:		
120 lb. Shear Grade	$\frac{1}{10}$	$\frac{3}{8}$
Wane	$\frac{1}{6}$ Width of any face	

OAK

Slope of Grain.—1 in 12.

Knots.—Maximum permissible size in inches:

NOMINAL WIDTH OF FACE, IN.	ON NARROW FACE, OR AT EDGE OF WIDE FACE, MIDDLE THIRD OF LENGTH	AT CENTER LINE OF WIDE FACE
2	$\frac{5}{8}$ in.	On	.....
3	1 in.	Narrow	.....
4	$1\frac{3}{8}$ in.	Face	.....
4	$\frac{3}{8}$ in.		$1\frac{1}{4}$ in.
6	$1\frac{1}{8}$ in.	At	2 in.
8	$1\frac{1}{2}$ in.	Edge	$2\frac{3}{4}$ in.
10	$1\frac{7}{8}$ in.	of	$3\frac{1}{2}$ in.
12	$2\frac{1}{4}$ in.	Wide	4 in.
14	$2\frac{3}{4}$ in.	Face	$4\frac{1}{2}$ in.
16	$2\frac{1}{2}$ in.		$4\frac{3}{4}$ in.

	WIDTH OF NARROW FACE When Green	When Seasoned
Shakes, Checks, Splits:		
120 lb. Shear Grade	$\frac{1}{4}$	$\frac{3}{4}$
Wane	$\frac{1}{6}$ Width of any face	

DENSE LONGLEAF SOUTHERN PINE

Slope of Grain.—1 in 10.

Knots.—Maximum permissible size in inches:

NOMINAL WIDTH OF FACE, IN.	ON NARROW FACE, OR AT EDGE OF WIDE FACE, MIDDLE THIRD OF LENGTH	AT CENTER LINE OF WIDE FACE
2	$\frac{7}{8}$ in.	On	.....
3	$1\frac{1}{2}$ in.	Narrow	.....
4	$1\frac{3}{4}$ in.	Face	.....
4	1 in.		$1\frac{3}{8}$ in.
6	$1\frac{1}{2}$ in.	At	$2\frac{3}{8}$ in.
8	2 in.	Edge	$3\frac{1}{2}$ in.
10	$2\frac{1}{2}$ in.	of	$4\frac{1}{2}$ in.
12	3 in.	Wide	$5\frac{1}{2}$ in.
14	$3\frac{1}{4}$ in.	Face	$5\frac{7}{8}$ in.
16	$3\frac{3}{8}$ in.		6 in.

	WIDTH OF NARROW FACE When Green	When Seasoned
Shakes, Checks, Splits:		
100 lb. Shear Grade	$\frac{1}{8}$	$\frac{4}{8}$
Wane	$\frac{1}{4}$ Width of any face	

CLOSE-GRAINED REDWOOD

Slope of Grain.—1 in 14.

Knots.—Maximum permissible size in inches:

NOMINAL WIDTH OF FACE, IN.	ON NARROW FACE, OR AT EDGE OF WIDE FACE, MIDDLE THIRD OF LENGTH	AT CENTER LINE OF WIDE FACE
2	$\frac{1}{2}$ in.	On	.....
3	$\frac{7}{8}$ in.	Narrow	.....
4	$1\frac{3}{8}$ in.	Face	.....
4	$\frac{5}{8}$ in.		$1\frac{1}{8}$ in.
6	$\frac{5}{8}$ in.	At	$1\frac{5}{8}$ in.
8	$1\frac{1}{2}$ in.	Edge	$2\frac{1}{2}$ in.
10	$1\frac{1}{2}$ in.	of	$2\frac{5}{8}$ in.
12	$1\frac{5}{8}$ in.	Wide	$3\frac{5}{8}$ in.
14	2 in.	Face	$3\frac{5}{8}$ in.
16	$2\frac{1}{8}$ in.		$3\frac{5}{8}$ in.

	WIDTH OF NARROW FACE When Green	When Seasoned
Shakes, Checks, Splits:		
80 lb. Shear Grade	$\frac{1}{2}$	$\frac{2}{3}$
Wane	$\frac{1}{6}$ Width of any face	

1200 lb. *f* Structural Joist and Plank

PORT ORFORD CEDAR

Slope of Grain.—1 in 14.

Knots.—Maximum permissible size in inches:

NOMINAL WIDTH OF FACE, IN.	ON NARROW FACE, OR AT EDGE OF WIDE FACE, MIDDLE THIRD OF LENGTH	AT CENTER LINE OF WIDE FACE
2	½ in.	On
3	⅝ in.	Narrow
4	1⅛ in.	Face
4	⅝ in.	1⅛ in.
6	⅞ in.	At 1⅝ in.
8	1⅛ in.	Edge 2¼ in.
10	1⅜ in.	of 2⅞ in.
12	1⅝ in.	Wide 3⅜ in.
14	2 in.	Face 3⅞ in.
16	2⅜ in.	3⅝ in.

	WIDTH OF NARROW FACE When Green	When Seasoned
Shakes, Checks, Splits: 100 lb. Shear Grade	1/6	1/4
Wane	1/6 Width of any face	

DOUGLAS FIR (COAST REGION)

Slope of Grain.—1 in 10.

Knots.—Maximum permissible size in inches:

NOMINAL WIDTH OF FACE, IN.	ON NARROW FACE, OR AT EDGE OF WIDE FACE, MIDDLE THIRD OF LENGTH	AT CENTER LINE OF WIDE FACE
2	⅞ in.	On
3	1¼ in.	Narrow
4	1⅝ in.	Face
4	1 in.	1⅜ in.
6	1½ in.	At 2⅛ in.
8	2 in.	Edge 3⅜ in.
10	2½ in.	of 4¼ in.
12	3 in.	Wide 5¼ in.
14	3½ in.	Face 5⅝ in.
16	3⅞ in.	6 in.

	WIDTH OF NARROW FACE When Green	When Seasoned
Shakes, Checks, Splits: 100 lb. Shear Grade	1/6	1/4
Wane	1/4 Width of any face	

DENSE SHORTLEAF SOUTHERN PINE

Slope of Grain.—1 in 8.

Knots.—Maximum permissible size in inches:

NOMINAL WIDTH OF FACE, IN.	ON NARROW FACE, OR AT EDGE OF WIDE FACE, MIDDLE THIRD OF LENGTH	AT CENTER LINE OF WIDE FACE
2	1 in.	On
3	1½ in.	Narrow
4	2 in.	Face
4	1⅛ in.	2 in.
6	1⅝ in.	At 3 in.
8	2⅛ in.	Edge 3⅞ in.
10	2⅝ in.	of 4⅞ in.
12	3⅛ in.	Wide 5⅞ in.
14	3⅝ in.	Face 6⅞ in.
16	4 in.	6⅝ in.

	WIDTH OF NARROW FACE When Green	When Seasoned
Shakes, Checks, Splits: 100 lb. Shear Grade	1/16	1/9
Wane	1/4 Width of any face	

CLOSE-GRAINED REDWOOD

Slope of Grain.—1 in 12.

Knots.—Maximum permissible size in inches:

NOMINAL WIDTH OF FACE, IN.	ON NARROW FACE, OR AT EDGE OF WIDE FACE, MIDDLE THIRD OF LENGTH	AT CENTER LINE OF WIDE FACE
2	¾ in.	On
3	1⅛ in.	Narrow
4	1½ in.	Face
4	⅞ in.	1⅜ in.
6	1¼ in.	At 2⅛ in.
8	1⅝ in.	Edge 2⅞ in.
10	2 in.	of 3⅝ in.
12	2⅜ in.	Wide 4⅜ in.
14	2⅝ in.	Face 4⅞ in.
16	2⅞ in.	5 in.

	WIDTH OF NARROW FACE When Green	When Seasoned
Shakes, Checks, Splits: 70 lb. Shear Grade	1/4	1/9
Wane	1/8 Width of any face	

1100 lb. *f* Structural Joist and Plank

Port Orford Cedar

Slope of Grain.—1 in 12.

Knots.—Maximum permissible size in inches:

Nominal Width of Face, in.	On Narrow Face, or at Edge of Wide Face, Middle Third of Length	At Center Line of Wide Face
2	$\frac{5}{8}$ in. On Narrow Face	.....
3	1 in. On Narrow Face	.....
4	$1\frac{3}{8}$ in. On Narrow Face	.....
4	$\frac{3}{4}$ in. At Edge of Wide Face	$1\frac{1}{4}$ in.
6	$1\frac{5}{16}$ in. At Edge of Wide Face	2 in.
8	$1\frac{7}{8}$ in. At Edge of Wide Face	$2\frac{5}{8}$ in.
10	$1\frac{15}{16}$ in. At Edge of Wide Face	$3\frac{1}{4}$ in.
12	$2\frac{1}{16}$ in. At Edge of Wide Face	4 in.
14	$2\frac{9}{16}$ in. At Edge of Wide Face	$4\frac{1}{8}$ in.
16	$2\frac{7}{8}$ in. At Edge of Wide Face	$4\frac{5}{8}$ in.

	Width of Narrow Face When Green	When Seasoned
Shakes, Checks, Splits: 80 lb. Shear Grade	$\frac{1}{3}$	$\frac{4}{10}$
Wane	$\frac{1}{6}$ Width of any face	

Tidewater Red Cypress

Slope of Grain.—1 in 10.

Knots and Pech.—Maximum permissible size in inches:

Nominal Width of Face, in.	On Narrow Face, or at Edge of Wide Face, Middle Third of Length	At Center Line of Wide Face
2	$\frac{3}{4}$ in. On Narrow Face	.....
3	$1\frac{1}{4}$ in. On Narrow Face	.....
4	$1\frac{5}{8}$ in. On Narrow Face	.....
4	$\frac{3}{4}$ in. At Edge of Wide Face	$1\frac{5}{8}$ in.
6	$1\frac{3}{8}$ in. At Edge of Wide Face	$2\frac{1}{2}$ in.
8	$1\frac{7}{8}$ in. At Edge of Wide Face	$3\frac{1}{4}$ in.
10	$2\frac{3}{8}$ in. At Edge of Wide Face	$4\frac{1}{8}$ in.
12	$2\frac{3}{4}$ in. At Edge of Wide Face	5 in.
14	3 in. At Edge of Wide Face	$5\frac{5}{8}$ in.
16	$3\frac{1}{4}$ in. At Edge of Wide Face	$5\frac{3}{4}$ in.

	Width of Narrow Face When Green	When Seasoned
Shakes, Checks, Splits: 100 lb. Shear Grade	$\frac{1}{4}$	$\frac{1}{3}$
Wane	$\frac{1}{4}$ Width of any face	

Oak

Slope of Grain.—1 in 10.

Knots.—Maximum permissible size in inches:

Nominal Width of Face, in.	On Narrow Face, or at Edge of Wide Face, Middle Third of Length	At Center Line of Wide Face
2	$\frac{7}{8}$ in. On Narrow Face	.....
3	$1\frac{3}{8}$ in. On Narrow Face	.....
4	$1\frac{3}{4}$ in. On Narrow Face	.....
4	1 in. At Edge of Wide Face	$1\frac{3}{4}$ in.
6	$1\frac{1}{2}$ in. At Edge of Wide Face	$2\frac{5}{8}$ in.
8	2 in. At Edge of Wide Face	$3\frac{1}{2}$ in.
10	$2\frac{1}{2}$ in. At Edge of Wide Face	$4\frac{3}{8}$ in.
12	3 in. At Edge of Wide Face	$5\frac{1}{4}$ in.
14	$3\frac{1}{4}$ in. At Edge of Wide Face	$5\frac{7}{8}$ in.
16	$3\frac{1}{2}$ in. At Edge of Wide Face	$6\frac{1}{8}$ in.

	Width of Narrow Face When Green	When Seasoned
Shakes, Checks, Splits: 100 lb. Shear Grade	$\frac{4}{10}$	$\frac{4}{9}$
Wane	$\frac{1}{4}$ Width of any face	

1000 lb. *f* Structural Joist and Plank

Western Red Cedar

Slope of Grain.—1 in 14.

Knots.—Maximum permissible size in inches:

Nominal Width of Face, in.	On Narrow Face, or at Edge of Wide Face, Middle Third of Length	At Center Line of Wide Face
2	$\frac{5}{8}$ in. On Narrow Face	.....
3	$\frac{7}{8}$ in. On Narrow Face	.....
4	$1\frac{1}{8}$ in. On Narrow Face	.....
4	$\frac{5}{8}$ in. At Edge of Wide Face	$1\frac{3}{8}$ in.
6	$\frac{7}{8}$ in. At Edge of Wide Face	$1\frac{7}{8}$ in.
8	$1\frac{1}{8}$ in. At Edge of Wide Face	$2\frac{1}{2}$ in.
10	$1\frac{3}{8}$ in. At Edge of Wide Face	$2\frac{7}{8}$ in.
12	$1\frac{5}{8}$ in. At Edge of Wide Face	$3\frac{3}{8}$ in.
14	2 in. At Edge of Wide Face	$3\frac{5}{8}$ in.
16	$2\frac{1}{8}$ in. At Edge of Wide Face	$3\frac{7}{8}$ in.

	Width of Narrow Face When Green	When Seasoned
Shakes, Checks, Splits: 100 lb. Shear Grade	$\frac{1}{18}$	$\frac{1}{6}$
Wane	$\frac{1}{6}$ Width of any face	

= Extreme fiber stress in bending in pounds per square inch for continuously dry locations

1800 lb. *f* Structural Beams and Stringers

DENSE DOUGLAS FIR (COAST REGION)
DENSE DOUGLAS FIR (INLAND)

DENSE LONGLEAF SOUTHERN PINE
DENSE SHORTLEAF SOUTHERN PINE

Slope of Grain.—1 in 15. *Knots.*—Maximum permissible size in inches:

NOMINAL WIDTH OF FACE, IN.	ON NARROW FACE, MIDDLE THIRD OF LENGTH	AT CENTER LINE OF WIDE FACE
5	1½ in.	.....
6	1⅝ in.	.....
8	1⅝ in.	1⅞ in.
10	1⅞ in.	2⅛ in.
12	1⅞ in.	2⅜ in.
14	2⅛ in.	2⅝ in.
16	2¼ in.	3⅛ in.
18	.....	3⅜ in.
20	.....	3½ in.

	WIDTH OF NARROW FACE	
	When Green	When Seasoned
Shakes, Checks, Splits:		
120 lb. Shear Grade (Fir)	⅛	¼
120 lb. Shear Grade (Pine)	$\frac{1}{10}$	⅜
Wane	⅛ Width of any face	

1600 lb. *f* Structural Beams and Stringers

CLOSE-GRAINED DOUGLAS FIR (COAST REGION)

Slope of Grain.—1 in 15.

Knots.—Maximum permissible size in inches:

NOMINAL WIDTH OF FACE, IN.	ON NARROW FACE, MIDDLE THIRD OF LENGTH	AT CENTER LINE OF WIDE FACE
5	1½ in.	.....
6	1⅝ in.	.....
8	1⅝ in.	1⅞ in.
10	1⅞ in.	2⅛ in.
12	1⅞ in.	2⅜ in.
14	2⅛ in.	2⅝ in.
16	2¼ in.	3⅛ in.
18	.....	3⅜ in.
20	.....	3½ in.

	WIDTH OF NARROW FACE	
	When Green	When Seasoned
Shakes, Checks, Splits:		
100 lb. Shear Grade	⅛	¼
Wane	⅙ Width of any face	

DENSE LONGLEAF SOUTHERN PINE
DENSE SHORTLEAF SOUTHERN PINE

Slope of Grain.—1 in 12.

Knots.—Maximum permissible size in inches:

NOMINAL WIDTH OF FACE, IN.	ON NARROW FACE, MIDDLE THIRD OF LENGTH	AT CENTER LINE OF WIDE FACE
5	1½ in.	.....
6	1⅝ in.	.....
8	2⅛ in.	2⅜ in.
10	2⅜ in.	3⅛ in.
12	2⅝ in.	3⅝ in.
14	2⅞ in.	4 in.
16	3 in.	4⅜ in.
18	.....	4⅝ in.
20	.....	4⅞ in.

	WIDTH OF NARROW FACE	
	When Green	When Seasoned
Shakes, Checks, Splits:		
120 lb. Shear Grade	$\frac{1}{10}$	⅜
Wane	⅛ Width of any face	

CLOSE-GRAINED REDWOOD

Slope of Grain.—1 in 20.

Knots.—Maximum permissible size in inches:

NOMINAL WIDTH OF FACE, IN.	ON NARROW FACE, MIDDLE THIRD OF LENGTH	AT CENTER LINE OF WIDE FACE
5	⅜ in.	.....
6	⅜ in.	.....
8	½ in.	1⅛ in.
10	½ in.	1⅜ in.
12	⅝ in.	1⅝ in.
14	⅝ in.	1⅞ in.
16	¾ in.	2⅛ in.
18	.....	1 in.
20	.....	1 in.

	WIDTH OF NARROW FACE	
	When Green	When Seasoned
Shakes, Checks Splits: 80 lb. Shear Grade	⅛	¼
Wane	$\frac{1}{10}$ Width of any face	

1400 lb. *f* Structural Beams and Stringers

TIDEWATER RED CYPRESS

Slope of Grain.—1 in 16.

Knots and Peck.—Maximum permissible size in inches:

NOMINAL WIDTH OF FACE, IN.	ON NARROW FACE, MIDDLE THIRD OF LENGTH	AT CENTER LINE OF WIDE FACE
5	1 in.	.....
6	1 1/4 in.	.....
8	1 3/8 in.	1 1/2 in.
10	1 1/2 in.	1 5/8 in.
12	1 5/8 in.	2 1/4 in.
14	1 3/4 in.	2 1/4 in.
16	1 5/8 in.	2 3/8 in.
18	.....	2 5/8 in.
20	.....	3 in.

	WIDTH OF NARROW FACE When Green	When Seasoned
Shakes, Checks, Splits: 120 lb. Shear Grade	1/10	1/5
Wane	1/5 Width of any face	

OAK

Slope of Grain.—1 in 15.

Knots.—Maximum permissible size in inches:

NOMINAL WIDTH OF FACE, IN.	ON NARROW FACE, MIDDLE THIRD OF LENGTH	AT CENTER LINE OF WIDE FACE
5	1 1/4 in.	.....
6	1 1/2 in.	.....
8	1 3/4 in.	2 in.
10	2 in.	2 1/2 in.
12	2 1/4 in.	3 in.
14	2 1/2 in.	3 1/2 in.
16	2 1/2 in.	3 1/2 in.
18	.....	3 3/4 in.
20	.....	3 3/4 in.

	WIDTH OF NARROW FACE When Green	When Seasoned
Shakes, Checks, Splits: 120 lb. Shear Grade	1/4	1/3
Wane	1/5 Width of any face	

DENSE LONGLEAF SOUTHERN PINE

Slope of Grain.—1 in 10.

Knots.—Maximum permissible size in inches:

NOMINAL WIDTH OF FACE, IN.	ON NARROW FACE, MIDDLE THIRD OF LENGTH	AT CENTER LINE OF WIDE FACE
5	2 in.	.....
6	2 3/8 in.	.....
8	2 3/4 in.	3 1/4 in.
10	3 1/4 in.	4 in.
12	3 3/8 in.	4 7/8 in.
14	3 5/8 in.	5 1/4 in.
16	4 in.	5 5/8 in.
18	.....	5 5/8 in.
20	.....	6 1/4 in.

	WIDTH OF NARROW FACE When Green	When Seasoned
Shakes, Checks, Splits: 100 lb. Shear Grade	4/10	2/5
Wane	1/4 Width of any face	

CLOSE-GRAINED REDWOOD

Slope of Grain.—1 in 18.

Knots.—Maximum permissible size in inches:

NOMINAL WIDTH OF FACE, IN.	ON NARROW FACE, MIDDLE THIRD OF LENGTH	AT CENTER LINE OF WIDE FACE
5	7/8 in.	.....
6	1 1/8 in.	.....
8	1 1/4 in.	1 1/2 in.
10	1 3/8 in.	1 3/4 in.
12	1 1/2 in.	2 1/4 in.
14	1 5/8 in.	2 1/2 in.
16	1 3/4 in.	2 1/2 in.
18	.....	2 3/4 in.
20	.....	2 3/4 in.

	WIDTH OF NARROW FACE When Green	When Seasoned
Shakes, Checks, Splits: 80 lb. Shear Grade	1/5	2/5
Wane	1/5 Width of any face	

1200 lb. *f* Structural Beams and Stringers

DENSE SHORTLEAF SOUTHERN PINE

Slope of Grain.—1 in 8.

Knots.—Maximum permissible size in inches:

NOMINAL WIDTH OF FACE, IN.	ON NARROW FACE, MIDDLE THIRD OF LENGTH	AT CENTER LINE OF WIDE FACE
5	2 3/8 in.	.....
6	2 7/8 in.	.....
8	3 3/8 in.	3 7/8 in.
10	3 7/8 in.	4 7/8 in.
12	4 3/8 in.	5 3/4 in.
14	4 7/8 in.	6 3/4 in.
16	4 3/4 in.	6 3/4 in.
18	.....	7 1/8 in.
20	.....	7 1/2 in.

	WIDTH OF NARROW FACE When Green	When Seasoned
Shakes, Checks, Splits: 100 lb. Shear Grade	4/10	2/5
Wane	1/4 Width of any face	

CLOSE-GRAINED REDWOOD

Slope of Grain.—1 in 14.

Knots.—Maximum permissible size in inches:

NOMINAL WIDTH OF FACE, IN.	ON NARROW FACE, MIDDLE THIRD OF LENGTH	AT CENTER LINE OF WIDE FACE
5	1 1/2 in.	.....
6	1 3/4 in.	.....
8	2 1/8 in.	2 5/8 in.
10	2 3/8 in.	3 in.
12	2 5/8 in.	3 5/8 in.
14	2 3/4 in.	3 7/8 in.
16	3 in.	4 1/4 in.
18	.....	4 1/2 in.
20	.....	4 1/2 in.

	WIDTH OF NARROW FACE When Green	When Seasoned
Shakes, Checks, Splits: 70 lb. Shear Grade	1/4	1/3
Wane	1/5 Width of any face	

1100 lb. *f* Structural Beams and Stringers

PORT ORFORD CEDAR

Slope of Grain.—1 in 15.

Knots.—Maximum permissible size in inches:

Nominal Width of Face, in.	On Narrow Face, Middle Third of Length	At Center Line of Wide Face
5	1½ in.	.....
6	1⅞ in.	.....
8	1⅞ in.	2 in.
10	2 in.	2⅜ in.
12	2⅜ in.	3 in.
14	2⅞ in.	3½ in.
16	2⅞ in.	3⅝ in.
18	.....	3¾ in.
20	.....	3⅞ in.

	Width of Narrow Face	
	When Green	When Seasoned
Shakes, Checks, Splits:		
80 lb. Shear Grade	1/3	4/10
Wane	1/6 Width of any face	

TIDEWATER RED CYPRESS

Slope of Grain.—1 in 12.

Knots and Peck.—Maximum permissible size in inches:

Nominal Width of Face, in.	On Narrow Face, Middle Third of Length	At Center Line of Wide Face
5	1¾ in.	.....
6	2⅛ in.	.....
8	2⅞ in.	2⅞ in.
10	2⅞ in.	3⅜ in.
12	3⅜ in.	4⅛ in.
14	3⅞ in.	4¾ in.
16	3½ in.	5 in.
18	.....	5⅜ in.
20	.....	5⅝ in.

	Width of Narrow Face	
	When Green	When Seasoned
Shakes, Checks, Splits:		
100 lb. Shear Grade	1/4	3/8
Wane	1/8 Width of any face	

OAK

Slope of Grain.—1 in 10.

Knots.—Maximum permissible size in inches:

Nominal Width of Face, in.	On Narrow Face, Middle Third of Length	At Center Line of Wide Face
5	2 in.	.....
6	2⅛ in.	.....
8	2⅞ in.	3½ in.
10	3½ in.	4⅛ in.
12	3⅝ in.	5 in.
14	3¾ in.	5⅜ in.
16	4 in.	5⅞ in.
18	.....	6 in.
20	.....	6⅛ in.

	Width of Narrow Face	
	When Green	When Seasoned
Shakes, Checks, Splits:		
100 lb. Shear Grade	4/10	4/9
Wane	¼ Width of any face	

1000 lb. *f* Structural Beams and Stringers

WESTERN RED CEDAR

Slope of Grain.—1 in 18.

Knots.—Maximum permissible size in inches:

Nominal Width of Face, in.	On Narrow Face, Middle Third of Length	At Center Line of Wide Face
5	¾ in.	.....
6	1 in.	.....
8	1⅛ in.	1⅜ in.
10	1⅛ in.	1⅝ in.
12	1⅝ in.	2 in.
14	1⅞ in.	2⅛ in.
16	1⅝ in.	2⅛ in.
18	.....	2⅜ in.
20	.....	2½ in.

	Width of Narrow Face	
	When Green	When Seasoned
Shakes, Checks, Splits:		
100 lb. Shear Grade	1/8	1/8
Wane	⅛ Width of any face	

c = Compression stress parallel to grain in pounds per square inch for continuously dry locations.

1300 lb. c Structural Posts and Timbers

Dense Douglas Fir (Coast Region)
Dense Douglas Fir (Inland)
Dense Longleaf Southern Pine
Dense Shortleaf Southern Pine

Slope of Grain.—1 in 12.

Knots.—Maximum permissible size in inches:

Nominal Width of Face, in.	At any Point on any Face
5	1¼ in.
6	1½ in.
8	1⅞ in.
10	2⅜ in.
12	2⅞ in.
14	3⅜ in.
16	3⅞ in.
18	3½ in.
20	3¾ in.

	Width of Narrow Face When Green	When Seasoned
Shakes, Checks, Splits	1/16	½
Wane	⅛ Width of any face	

1200 lb. c Structural Posts and Timbers

Tidewater Red Cypress

Slope of Grain.—1 in 12.

Knots and Peck.—Maximum permissible size in inches:

Nominal Width of Face, in.	At any Point on any Face
5	⅞ in.
6	1⅛ in.
8	1½ in.
10	1⅞ in.
12	2¼ in.
14	2⅜ in.
16	2½ in.
18	2¾ in.
20	2¾ in.

	Width of Narrow Face When Green	When Seasoned
Shakes, Checks, Splits	½	⅛
Wane	⅛ Width of any face	

Close-Grained Douglas Fir (Coast Region)

Slope of Grain.—1 in 12.

Knots.—Maximum permissible size in inches:

Nominal Width of Face, in.	At any Point on any Face
5	1⅛ in.
6	1⅜ in.
8	1⅞ in.
10	2⅜ in.
12	2⅞ in.
14	3 in.
16	3¼ in.
18	3⅜ in.
20	3⅞ in.

	Width of Narrow Face When Green	When Seasoned
Shakes, Checks, Splits	1/10	½
Wane	⅛ Width of any face	

Dense Longleaf Southern Pine
Dense Shortleaf Southern Pine

Slope of Grain.—1 in 10.

Knots.—Maximum permissible size in inches:

Nominal Width of Face, in.	At any Point on any Face
5	1⅛ in.
6	1⅜ in.
8	2⅜ in.
10	3 in.
12	3⅝ in.
14	3⅞ in.
16	4¼ in.
18	4⅜ in.
20	4⅝ in.

	Width of Narrow Face When Green	When Seasoned
Shakes, Checks, Splits	1/16	⅛
Wane	⅛ Width of any face	

Close-Grained Redwood

Slope of Grain.—1 in 14.

Knots.—Maximum permissible size in inches:

Nominal Width of Face, in.	At any Point on any Face
5	¾ in.
6	⅞ in.
8	1⅛ in.
10	1⅜ in.
12	1⅝ in.
14	2 in.
16	2⅛ in.
18	2⅜ in.
20	2⅝ in.

	Width of Narrow Face When Green	When Seasoned
Shakes, Checks, Splits	⅛	1/16
Wane	⅛ Width of any face	

1100 lb. c Structural Posts and Timbers

DOUGLAS FIR (COAST REGION)

Slope of Grain.—1 in 10.

Knots.—Maximum permissible size in inches:

NOMINAL WIDTH OF FACE, IN.	AT ANY POINT ON ANY FACE
5	1½ in.
6	1½ in.
8	2 in.
10	2½ in.
12	3 in.
14	3½ in.
16	3½ in.
18	3⅞ in.
20	3⅞ in.

	WIDTH OF NARROW FACE	
	When Green	When Seasoned
Shakes, Checks, Splits	1/10	½
Wane	⅛ Width of any face	

OAK

Slope of Grain.—1 in 14.

Knots.—Maximum permissible size in inches:

NOMINAL WIDTH OF FACE, IN.	AT ANY POINT ON ANY FACE
5	⅞ in.
6	1 in.
8	1⅜ in.
10	1⅞ in.
12	2⅛ in.
14	2⅜ in.
16	2⅜ in.
18	2⅝ in.
20	2⅝ in.

	WIDTH OF NARROW FACE	
	When Green	When Seasoned
Shakes, Checks, Splits	⅓	⅜
Wane	⅛ Width of any face	

CLOSE-GRAINED REDWOOD

Slope of Grain.—1 in 12.

Knots.—Maximum permissible size in inches:

NOMINAL WIDTH OF FACE, IN.	AT ANY POINT ON ANY FACE
5	1⅛ in.
6	1⅛ in.
8	1⅜ in.
10	2¼ in.
12	2⅝ in.
14	2⅝ in.
16	3⅛ in.
18	3⅛ in.
20	3½ in.

	WIDTH OF NARROW FACE	
	When Green	When Seasoned
Shakes, Checks, Splits	⅜	½
Wane	⅛ Width of any face	

1000 lb. *c* Structural Posts and Timbers

PORT ORFORD CEDAR

Slope of Grain.—1 in 14.

Knots.—Maximum permissible size in inches:

NOMINAL WIDTH OF FACE, IN.	AT ANY POINT ON ANY FACE
5	$\frac{7}{8}$ in.
6	1 in.
8	$1\frac{1}{8}$ in.
10	$1\frac{1}{8}$ in.
12	2 in.
14	$2\frac{1}{8}$ in.
16	$2\frac{1}{8}$ in.
18	$2\frac{1}{8}$ in.
20	$2\frac{1}{8}$ in.

	WIDTH OF NARROW FACE	
	When Green	When Seasoned
Shakes, Checks, Splits	$\frac{1}{3}$	$\frac{2}{3}$
Wane	$\frac{1}{8}$ Width of any face	

TIDEWATER RED CYPRESS

Slope of Grain.—1 in 10.

Knots and Peck.—Maximum permissible size in inches:

NOMINAL WIDTH OF FACE, IN.	AT ANY POINT ON ANY FACE
5	$1\frac{1}{8}$ in.
6	$1\frac{1}{8}$ in.
8	$2\frac{1}{8}$ in.
10	$3\frac{1}{4}$ in.
12	$3\frac{1}{4}$ in.
14	4 in.
16	$4\frac{1}{8}$ in.
18	$4\frac{1}{2}$ in.
20	5 in.

	WIDTH OF NARROW FACE	
	When Green	When Seasoned
Shakes, Checks, Splits	$\frac{4}{5}$	$\frac{5}{6}$
Wane	$\frac{1}{6}$ Width of any face	

OAK

Slope of Grain.—1 in 12.

Knots.—Maximum permissible size in inches:

NOMINAL WIDTH OF FACE, IN.	AT ANY POINT ON ANY FACE
5	$1\frac{1}{2}$ in.
6	$1\frac{1}{2}$ in.
8	2 in.
10	$2\frac{1}{2}$ in.
12	3 in.
14	$3\frac{1}{2}$ in.
16	$3\frac{1}{2}$ in.
18	$3\frac{1}{2}$ in.
20	$3\frac{1}{2}$ in.

	WIDTH OF NARROW FACE	
	When Green	When Seasoned
Shakes, Checks, Splits	$\frac{4}{10}$	$\frac{3}{5}$
Wane	$\frac{1}{6}$ Width of any face	

DENSE LONGLEAF SOUTHERN PINE

Slope of Grain.—1 in 8.

Knots.—Maximum permissible size in inches:

NOMINAL WIDTH OF FACE, IN.	AT ANY POINT ON ANY FACE
5	$2\frac{1}{8}$ in.
6	$2\frac{1}{8}$ in.
8	$3\frac{3}{8}$ in.
10	$4\frac{1}{8}$ in.
12	5 in.
14	$5\frac{3}{8}$ in.
16	$5\frac{3}{8}$ in.
18	$6\frac{1}{8}$ in.
20	$6\frac{3}{8}$ in.

	WIDTH OF NARROW FACE	
	When Green	When Seasoned
Shakes, Checks, Splits	$\frac{1}{2}$	$\frac{5}{6}$
Wane	$\frac{1}{4}$ Width of any face	

CLOSE-GRAINED REDWOOD

Slope of Grain.—1 in 10.

Knots.—Maximum permissible size in inches:

NOMINAL WIDTH OF FACE, IN.	AT ANY POINT ON ANY FACE
5	$1\frac{3}{8}$ in.
6	$1\frac{3}{8}$ in.
8	$2\frac{3}{8}$ in.
10	$2\frac{7}{8}$ in.
12	$3\frac{3}{8}$ in.
14	$3\frac{3}{8}$ in.
16	$4\frac{3}{8}$ in.
18	$4\frac{3}{8}$ in.
20	$4\frac{3}{8}$ in.

	WIDTH OF NARROW FACE	
	When Green	When Seasoned
Shakes, Checks, Splits	$\frac{3}{7}$	$\frac{1}{2}$
Wane	$\frac{1}{8}$ Width of any face	

900 lb. *c* Structural Posts and Timbers

PORT ORFORD CEDAR

Slope of Grain.—1 in 12.

Knots.—Maximum permissible size in inches:

NOMINAL WIDTH OF FACE, IN.	AT ANY POINT ON ANY FACE
5	1½ in.
6	1½ in.
8	2 in.
10	2½ in.
12	3 in.
14	3½ in.
16	3½ in.
18	3¾ in.
20	3¾ in.

	WIDTH OF NARROW FACE	
	When Green	When Seasoned
Shakes, Checks, Splits	1/10	½
Wane	1/6 Width of any face	

DENSE SHORTLEAF SOUTHERN PINE

Slope of Grain.—1 in 6.

Knots.—Maximum permissible size in inches:

NOMINAL WIDTH OF FACE, IN.	AT ANY POINT ON ANY FACE
5	2⅜ in.
6	2⅞ in.
8	3⅝ in.
10	4⅜ in.
12	5⅛ in.
14	6⅛ in.
16	6½ in.
18	7 in.
20	7½ in.

	WIDTH OF NARROW FACE	
	When Green	When Seasoned
Shakes, Checks, Splits	⅛	¾
Wane	¼ Width of any face	

800 lb. *c* Posts and Timbers

WESTERN RED CEDAR

Slope of Grain.—1 in 14.

Knots.—Maximum permissible size in inches:

NOMINAL WIDTH OF FACE, IN.	AT ANY POINT ON ANY FACE
5	1⅜ in.
6	1⅝ in.
8	1⅞ in.
10	1⅞ in.
12	1⅞ in.
14	1⅞ in.
16	2 in.
18	2⅛ in.
20	2⅛ in.

	WIDTH OF NARROW FACE	
	When Green	When Seasoned
Shakes, Checks; Splits	1/10	⅛
Wane	⅛ Width of any face	

APPENDIX

The following three tables summarizing standard sizes, stress-grades, and working stresses for joist and plank, beams and stringers, and posts and timbers are appended for information. They offer a valuable key to different structural grades.

TABLE I.—STANDARD SIZES, STRESS-GRADES, AND WORKING STRESSES FOR STRUCTURAL JOIST AND PLANK.

Standard Sizes

Nominal thicknesses	2, 3, and 4 in.
Permissible minimum rough thicknesses in not to exceed 20 per cent of pieces in any one shipment:	
2 in.	$\frac{1}{8}$ in. off
3 and 4 in.	$\frac{3}{16}$ in. off
Dressed thicknesses, S1S or S2S	$\frac{3}{8}$ in. off
Nominal widths	4 in. and wider
Permissible minimum rough widths in not to exceed 20 per cent of pieces in any one shipment:	
4 and 6 in.	$\frac{3}{16}$ in. off
8 in. and wider	$\frac{1}{4}$ in. off
Dressed widths, S1E or S2E:	
4 and 6 in.	$\frac{1}{8}$ in. off
8 in. and wider	$\frac{1}{2}$ in. off

Standard Stress-Grades and Working Stresses

(Continuously Dry Locations)

GRADES AND SPECIES	FIBER STRESS IN BENDING OR TENSION, PSI.	MAXIMUM HORIZONTAL SHEAR, PSI.	COMPRESSION PERPENDICULAR TO GRAIN, PSI.	MODULUS OF ELASTICITY, PSI.
1800 lb. *f* Dense Douglas Fir (Coast and Inland)	1800	120	380	1 600 000
1800 lb. *f* Dense Longleaf or Dense Shortleaf Southern Pine	1800	120	380	1 600 000
1600 lb. *f* Close-Grained Douglas Fir (Coast)	1600	100	345	1 600 000
1600 lb. *f* Dense Longleaf or Dense Shortleaf Southern Pine	1600	120	380	1 600 000
1600 lb. *f* Close-Grained Redwood	1600	80	267	1 200 000
1400 lb. *f* Tidewater Red Cypress	1400	120	300	1 200 000
1400 lb. *f* Oak	1400	120	500	1 500 000
1400 lb. *f* Dense Longleaf Southern Pine	1400	100	380	1 600 000
1400 lb. *f* Close-Grained Redwood	1400	80	267	1 200 000
1200 lb. *f* Port Orford Cedar	1200	100	250	1 200 000
1200 lb. *f* Douglas Fir (Coast)	1200	100	325	1 600 000
1200 lb. *f* Dense Shortleaf Southern Pine	1200	100	380	1 600 000
1200 lb. *f* Close-Grained Redwood	1200	70	267	1 200 000
1100 lb. *f* Port Orford Cedar	1100	80	250	1 200 000
1100 lb. *f* Tidewater Red Cypress	1100	100	300	1 200 000
1100 lb. *f* Oak	1100	100	500	1 500 000
1000 lb. *f* Western Red Cedar	1000	100	200	1 000 000

TABLE II.—STANDARD SIZES, STRESS-GRADES, AND WORKING STRESSES FOR STRUCTURAL BEAMS AND STRINGERS.

Standard Sizes

Nominal thicknesses	5 in. and thicker
Permissible minimum rough thicknesses in not to exceed 20 per cent of pieces in any one shipment:	
5 and 6 in.	$\frac{3}{16}$ in. off
8 in. and wider	$\frac{1}{4}$ in. off
Dressed thicknesses, S1S or S2S	$\frac{1}{2}$ in. off
Nominal widths	8 in. and wider
Permissible minimum rough widths in not to exceed 20 per cent of pieces in any one shipment	$\frac{1}{4}$ in. off
Dressed widths, S1E or S2E	$\frac{1}{2}$ in. off

Standard Stress-Grades and Working Stresses

(Continuously Dry Locations)

GRADES AND SPECIES	FIBER STRESS IN BENDING OR TENSION, PSI.	MAXIMUM HORIZONTAL SHEAR, PSI.	COMPRESSION PERPENDICULAR TO GRAIN, PSI.	MODULUS OF ELASTICITY, PSI.
1800 lb. *f* Dense Douglas Fir (Coast and Inland)	1800	120	380	1 600 000
1800 lb. *f* Dense Longleaf or Dense Shortleaf Southern Pine	1800	120	380	1 600 000
1600 lb. *f* Close-Grained Douglas Fir (Coast)	1600	100	345	1 600 000
1600 lb. *f* Dense Longleaf or Dense Shortleaf Southern Pine	1600	120	380	1 600 000
1600 lb. *f* Close-Grained Redwood	1600	80	267	1 200 000
1400 lb. *f* Tidewater Red Cypress	1400	120	300	1 200 000
1400 lb. *f* Oak	1400	120	500	1 500 000
1400 lb. *f* Dense Longleaf Southern Pine	1400	100	380	1 600 000
1400 lb. *f* Close-Grained Redwood	1400	80	267	1 200 000
1200 lb. *f* Dense Shortleaf Southern Pine	1200	100	380	1 600 000
1200 lb. *f* Close-Grained Redwood	1200	70	267	1 200 000
1100 lb. *f* Port Orford Cedar	1100	80	250	1 200 000
1100 lb. *f* Tidewater Red Cypress	1100	100	300	1 200 000
1100 lb. *f* Oak	1100	100	500	1 500 000
1000 lb. *f* Western Red Cedar	1000	100	200	1 000 000

TABLE III.—STANDARD SIZES, STRESS-GRADES, AND WORKING STRESSES FOR STRUCTURAL POSTS AND TIMBERS.

Standard Sizes

Nominal thicknesses	5 in. and thicker
Permissible minimum rough thicknesses in not to exceed 20 per cent of pieces in any one shipment:	
5 and 6 in.	$\frac{3}{16}$ in. off
8 in. and wider	$\frac{1}{4}$ in. off
Dressed thicknesses, S1S or S2S	$\frac{1}{2}$ in. off
Nominal widths	5 in. and wider
Permissible minimum rough widths in not to exceed 20 per cent of pieces in any one shipment:	
5 and 6 in.	$\frac{3}{16}$ in. off
8 in. and wider	$\frac{1}{4}$ in. off
Dressed widths, S1E or S2E	$\frac{1}{2}$ in. off

Standard Stress-Grades and Working Stresses

(Continuously Dry Locations)

GRADES AND SPECIES	COMPRESSION PARALLEL TO GRAIN, SHORT COLUMNS, PSI.	
1300 lb. *c* Dense Douglas Fir (Coast and Inland)	1300	
1300 lb. *c* Dense Longleaf or Dense Shortleaf Southern Pine	1300	
1200 lb. *c* Tidewater Red Cypress	1200	
1200 lb. *c* Close-Grained Douglas Fir (Coast)	1200	
1200 lb. *c* Dense Longleaf or Dense Shortleaf Southern Pine	1200	
1200 lb. *c* Close-Grained Redwood	1200	
1100 lb. *c* Douglas Fir (Coast)	1100	For stresses in compression
1100 lb. *c* Oak	1100	perpendicular to grain and
1100 lb. *c* Close-Grained Redwood	1100	modulus of elasticity, see
1000 lb. *c* Port Orford Cedar	1000	values in Table II for
1000 lb. *c* Tidewater Red Cypress	1000	Beams and Stringers.
1000 lb. *c* Oak	1000	
1000 lb. *c* Dense Longleaf Southern Pine	1000	
1000 lb. *c* Close-Grained Redwood	1000	
900 lb. *c* Port Orford Cedar	900	
900 lb. *c* Dense Shortleaf Southern Pine	900	
800 lb. *c* Western Red Cedar	800	

The detailed reasoning basic to these grades will be found by a study of a report of the U. S. Forest Products Laboratory, U. S. Department of Agriculture *Miscellaneous Publication No. 185*, entitled "Guide to the Grading of Structural Timbers and the Determination of Working Stresses," February, 1934. Reference should also be made to the "Working Stresses" appearing in the Appendix to the Standard Specifications for Structural Wood Joist and Plank, Beams and Stringers, and Posts and Timbers (A.S.T.M. Designation: D 245 – 33) of the American Society for Testing Materials, 1933 Book of A.S.T.M. Standards, Part II, p. 384.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: 04a-1927

Standard Methods of

TESTING SMALL CLEAR SPECIMENS OF TIMBER[1]

A.S.T.M. Designation: D 143 – 27

ADOPTED, 1927.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 143; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

The everyday use of timber for multitudinous purposes makes manifest a continual need of data on its mechanical properties. The great variety of species, the variability of the material, the continually changing conditions of supply, the many factors affecting test results, all combine to make the technique of testing wood unique in its complexity.

In the preparation of this procedure for testing small clear specimens, consideration was given both to the desirability of adopting methods which would yield results comparable to those already available and to the possibility of embodying such improvements as experience has shown desirable. In view of the many thousands of tests made under a single comprehensive plan by the U. S. Forest Service, the Forestry Branch of the Department of the Interior, Canada, and other similar organizations, the procedure naturally conforms closely to the methods used by these institutions. This procedure is the outgrowth of a study of both American and European experience and methods. Its general adoption will tend toward a world-wide unification of results, permitting an interchange and correlation of data, and will establish the basis for a cumulative body of fundamental information on the timber species of the world.

These methods represent the entire procedure from selection of the trees to the manipulation of the test, thus controlling factors, such as the size and proportion of test specimens and rate of loading, which may influence results. No attempt has been made to cover methods of computation and analysis, as these questions may be considered independently at any time. Such sample data and computation sheets and cards have been incorporated, however, as were thought to be of assistance to the investigator in systematizing records.

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[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-7 on Timber.

[2] Prior to adoption as standard, these methods were published as tentative from 1922 to 1927, being revised in 1924.

Purpose of Tests

1. Tests on small clear specimens of wood are made to afford:

(*a*) Data for comparing the mechanical properties of various species.

(*b*) Data for the establishment of correct strength functions which, in conjunction with results of tests of timbers in structural sizes,[3] afford the basis for fixing allowable stresses.

(*c*) Data upon which to determine the influence on the mechanical properties of such factors as density, locality of growth, position in cross-section, height of timber in the tree, change of properties with seasoning, and change from sapwood to heartwood.

Kind of Tests

2. The principal mechanical tests are static bending, compression parallel to grain, impact bending, compression perpendicular to grain, hardness, shear parallel to grain,[4] tension perpendicular to grain, cleavage, and tension parallel to grain. These tests are made on both green and air-dry material as specified in these methods. In addition, such physical properties as specific gravity, radial shrinkage, tangential shrinkage, and shrinkage in volume are determined.

COLLECTION OF MATERIAL

Selection

Authentic Identification

3. The material shall be from trees selected in the forest by one qualified to identify the species.

Selection and Number of Trees

4. For each species to be tested from a given locality, at least five representative trees of merchantable size and of approximately average age shall be selected.

Selection and Number of Bolts

5. The material from five trees of each species from a given locality shall be selected as follows:

(*a*) From one tree, the entire merchantable bole, if not over 16 ft. in length;[5] if over 16 ft. in length, four 4-ft. sections or bolts, the first to be taken at the stump, the second and third at distances of one-eighth and three-eighths, respectively, of the merchantable length above the stump or as near thereto as possible; and the fourth at the top of the merchantable length.

(*b*) From two trees, the 8-ft. section next above the 8-ft. butt log.

(*c*) From two trees, the 4-ft. section next above the 12-ft. butt log.

Substitution of Flitches for Bolts

6. In cases where the logs or bolts are over 36 in. in diameter, a single flitch 6 in. in thickness, taken through the pith in a north and south direction and representing the full diameter of the log, may be substituted, in the same length, for the full log or bolt specified in Section 5.

Selection from Small Trees

7. When the trees are relatively small in diameter, material in addition to that provided for in Sections 5 and 6 shall be collected, whenever possible, to insure a sufficient number of tests to give a good average for the species.

Field Marking

Tree Designation

8. Each tree shall be given an arabic number, the numbering in any given

[3] See Standard Methods of Static Tests of Timbers in Structural Sizes (A.S.T.M. Designation: D 198), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

[4] The test for shearing strength perpendicular to the grain (sometimes termed "vertical shear") is not included as one of the principal mechanical tests since in such a test the strength is limited by the shearing resistance parallel to the grain.

[5] This tree is intended to furnish test material for studying, among other things, the variation of properties with height in trees and provides for this purpose a 4-ft. section from different heights. Experience has shown that four sections or bolts, each 4 ft. in length, will usually be sufficient for studying the influence of height in tree on the mechanical properties.

shipment to be consecutive for trees of a given species.

Bolt Designation

9. Each 4 ft. of length of a tree or log shall be considered a "bolt." Bolts shall be designated by small letters, beginning with *a* for the 4-ft. section next above the stump. Bolt letters, given a shipment number or other designation.

Field Descriptions

Field Descriptions

13. (*a*) Complete field notes describing the material shall be fully and carefully made by the collector. These

TABLE I.—SHIPMENT DESCRIPTION FIELD NOTES.

Project No., *124* — Shipment No., *323*
Tree No., *10* — Species, *Lodgepole pine*
Locality cut: State, *Montana* — County, *Jefferson*
Township, *6 N., R. 7 W., M. P. M.* — Date cut, *Oct. 10, 1914*
Slope, *Moderate* — Absolute elevation, *6600 ft.* — Aspect, *southeasterly*
Undergrowth, *None* — Soil, *Deep, fresh-decomposed granite*
Height, *73.9 ft.* — Seedling or sprout, *Seedling* — Age, *140 yr.*
Crown, *Length 25 ft., width 12 ft.* — D.B.H. *11.1 in.*
Stem, *Sound, smooth, clear, length 20 ft.* — Date sawed, *Oct. 10, 1914*
How and when transported from woods, *By sleigh and wagon, Oct. 13 to 15, 1914*

LIST OF SPECIMENS SAWED.

Log Number	Bolts	Length, ft.	Diameter Inside Bark		Volume	Footage, b. m.	Weight, Oct. 15, lb.
			Butt, in.	Top, in.			
1	bcd	12.1	11	9.6	..	..	336
2	efg	12.1	9.6	8.9	..	..	289
3	hij	12.1	8.9	7.7	..	..	242
4	klm	12.1	7.7	6.1	..	..	176
5	nop	12.1	6.1	3.1	..	..	95

REMARKS: *Stump height, 1.0 ft. Butt off on account of frost crack 3.0 ft. Length of top above last section taken, 9.4 ft. Total age, 140 yr. Tree was dominant throughout life. Rate of growth, regular and good average for Site I. Lower slope type, although of more than average height for the age and diameter found on poorer sites.*

therefore, indicate position with respect to height in tree.

Manner of Marking

10. The tree number and bolt designation shall be plainly marked upon each log selected by the collector. Thus the 16-ft. butt log of Tree No. 2 would be designated *2abcd*. Steel dies are recommended for marking the butt end of the logs.

Indication of Cardinal Point

11. The north side of each log shall be indicated in some convenient manner.

Shipment Number

12. All material collected from a given locality and shipped at one time shall be notes shall, in general, supply data as outlined in Table I.

(*b*) Photographs of the standing trees selected shall be taken.

Preparation for Shipment

Preparation for Shipment

14. (*a*) The bark will be left on each log, and care shall be taken to keep the bark intact. The ends of the logs shall be carefully painted to retard or prevent end drying and end checking.

(*b*) Record shall be made of the shipment routing, bill of lading, kind of shipment, date of shipment, and condition of material when shipped.

(*c*) Record shall also be made of date of receipt of shipment at destination, its condition, and method of storage.

to artificial heat. In addition they should preferably be protected from the sun.

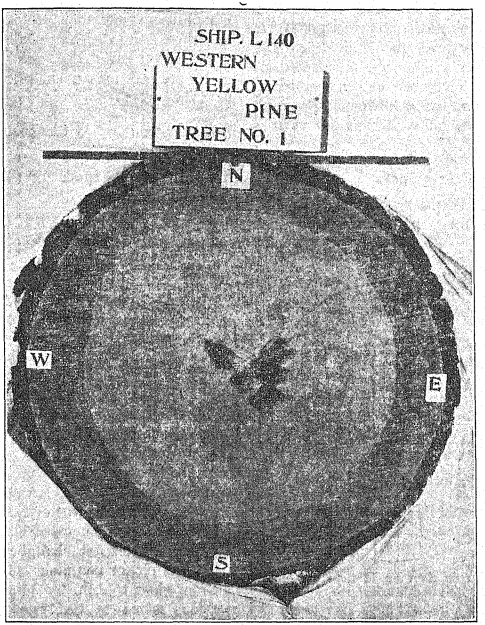


FIG. 1.—Section of Log Selected for Test Material.

DISPOSITION AT DESTINATION

Storage of Logs at Destination

Storage of Logs

15. Material shall not be kept in the bolt or log form long enough to permit damage by checks, decay, stains, or insect attack. The logs shall be piled on skids, free from contact with the soil, and shall not be stored where subjected

Photographing, Sawing and Final Marking

Photographing Ends of Bolts

16. The top end of each *d* or *c-d* bolt shall be photographed. It is suggested that a rule be so placed on the log as to indicate the scale of the photograph and that the cardinal points be indicated on the cross-section. Fig. 1 shows a photograph of this kind.

Sawing of Bolts

17. (*a*) All bolts shall be marked on the top end into 2½ by 2½-in. squares as shown in Fig. 2, and sawed into nominal 2½ by 2½-in. sticks. The letters *N*, *E*, *S*, and *W* indicate the cardinal points.

(*b*) When flitches are substituted for bolts (Section 6), the same general marking and numbering scheme of Paragraph (*a*) shall be followed in so far as it is applicable.

Marking of Test Sticks

18. All test sticks shall bear the shipment number, the tree number, stick

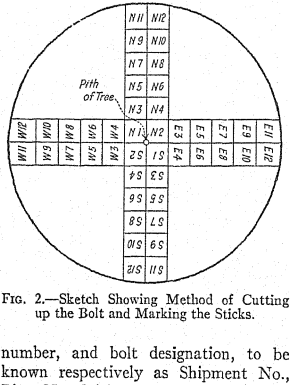


FIG. 2.—Sketch Showing Method of Cutting up the Bolt and Marking the Sticks.

number, and bolt designation, to be known respectively as Shipment No., Piece No., Stick No., and mark. Thus, 400-1-*N*4*d* represents Stick *N*4 of Bolt *d*, Tree 1, Shipment 400.

Matching for Air-dry Tests

Composition Bolts

19. In the tests made to determine the effect of air-drying on strength, the sticks from the *c* bolt shall be interchanged with those of the *d* bolt of the same tree to form two composition bolts, each composition bolt being complete and being made of equal portions of *c* and *d* bolts.[6] The sticks from one of these composition bolts shall be tested green and those from the other shall be tested after air-drying. The sticks tested either green or air-dry shall be regarded as if they were from the same bolt.

Schedule for Forming Composition Bolts

20. The division of green and air-dry sticks shall be made according to the following schedule in which the numbers refer to stick numbers:

Composition Bolt to be Tested Green:
Lower bolt *c*..... 1 4, 5 8, 9
Upper bolt *d*..... 2, 3 6, 7 10, etc.

Composition Bolt to be Air-Dried and Tested:
Lower bolt *c*..... 2, 3 6, 7 10, etc.
Upper bolt *d*..... 1 4, 5 8, 9

DISPOSITION OF STICKS

Green Material

21. The sticks (2½ by 2½ in. by 4 ft.) to be tested green shall be kept in an unseasoned condition while awaiting preparation for test by being stored in a framed pit or other suitable container, where they shall be close piled and covered with damp sawdust or in some other suitable manner. As material is required for test, it shall be removed from this pit, or container, surfaced on all four sides to 2 in. by 2 in. in cross-section, sawed to test size and kept covered with a damp cloth, in an air-tight container at a temperature of about 70 F. (as nearly as practicable) until the time of test. Care shall be taken to avoid as much as possible the storage of green material in any form. Sticks to be tested in a green condition should usually not be sawed from the log form in quantities greater than is required to meet the testing demands for from a few

[6] The 8-ft section (*c* and *d* bolts) from each of the two trees provided for under Section 5 (*b*) is intended for tests to determine the effect of air-drying on strength.

days to not more than two weeks, depending on the prevailing conditions.

Air-Dry Material

22. (*a*) The ends of the sticks to be air-dried (2½ by 2½ in. by 4 ft.) shall be dipped in melted paraffin or other substance suitable to retard checking. The material shall be piled so as to have a space of at least ¾ in. on each side of each stick to permit circulation of air. The material shall be stored in a place allowing free access of air, but protected from sunshine, rain, snow, and moisture from the ground. The sticks in drying shall not be subjected to artificial heat.

(*b*) All of the sticks from each composite bolt to be air-dried shall be weighed when stored and at sufficiently frequent intervals thereafter to get accurate data on the progress of seasoning. No material shall be considered thoroughly air-dried and properly conditioned for testing until practically constant weight has been reached. (Wood absorbs and gives off moisture with changing atmospheric conditions, consequently it never comes to absolutely constant weight.)

(*c*) When the material has reached equilibrium, moisture sections 1 in. in length shall be taken from about 10 per cent of the sticks to determine the actual moisture content. These moisture specimens shall be cut not less than 1 ft. from the ends of the sticks, and in such a way as to prevent any appreciable loss of material for testing. When thoroughly air-dry, the material shall be surfaced on four sides to 2 in. by 2 in. in cross-section and tested.

ORDER, SELECTION, AND NUMBER OF TESTS

Order of Tests

Order of Tests

23. The order of tests in all cases shall be such as to eliminate as far as possible from the comparisons the effect of changes in the specimen due to such factors as storage and weather conditions.

Selection of Specimens

Preference in Selecting Specimens

24. In case the material from a given bolt should be insufficient to furnish all the test specimens hereinafter required, the preferential order of mechanical tests to be used in selecting specimens shall be as follows: Static bending, compression parallel to grain, impact bending, compression perpendicular to grain, hardness, shear parallel to grain, tension perpendicular to grain, cleavage, and tension parallel to grain.

Test Pieces from Bending Specimens After Failure

25. In many cases where a shortage of material exists, certain test specimens may be taken from the uninjured portion of the static and impact bending specimens remaining after test, provided proper care is used in the selection.

Quality of Test Material

26. Only clear straight-grained material, free from decay and other defects, shall be used for the tests. However, small knots and other similar defects may be admitted in such specimens as static bending when their location is such that it is certain they will not in any way influence the failure or otherwise affect the strength of the specimen.

Number of Tests for Each Bolt

Static Bending

27. One static bending specimen shall be taken from each pair of sticks. A pair consists of two adjacent sticks equidistant from the pith, as *W*3 and *W*4, Fig. 2. In the composite bolts tested to afford a comparison of the

strength of green and air-dry material, the pair of sticks shall be constituted as above, except that the sticks in this case will be from different bolts. Thus *W3d* and *W4c* constitute one pair of sticks to be tested green, and *W3c* and *W4d* the corresponding pair to be tested air-dry (Section 20).

Compression Parallel to Grain

28. One compression-parallel-to-grain specimen shall be taken from each stick. Load-compression curves shall be taken on 20 per cent of the specimens.

Compression Perpendicular to Grain

30. One compression-perpendicular-to-grain specimen shall be taken from each of 50 per cent of the sticks selected for static bending.

Hardness

31. One hardness specimen shall be taken from each of the other 50 per cent of the static-bending sticks.

Shear Parallel to Grain

32. Eight shear-parallel-to-grain specimens shall be selected from the unused

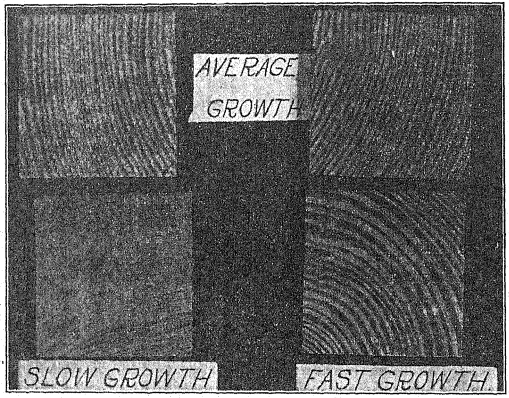


FIG. 3.—Cross-Sections of Bending Specimens Showing Different Rates of Growth of Longleaf Pine (2 by 2-in. Specimens).

Impact Bending

29. Eight impact-bending specimens shall be taken from each bolt, selection being made from the sticks remaining after obtaining the static bending tests. Two of the specimens shall be selected from near the pith, two from near the periphery, and four which are representative of the cross-section.

portion or ends of four sticks from which bending specimens have been selected. Two specimens shall be taken from near the pith, two from near the periphery, and four that are representative of the average growth of the cross-section of the bolt. One of each pair of specimens from the same stick shall be tested in radial shear (surface of failure radial)

and the other in tangential shear (surface of failure tangential).

Tension Perpendicular to Grain

33. Eight tension-perpendicular-to-grain specimens shall be selected from four sticks in a manner similar to that for shear (Section 32). One of each pair of specimens from the same stick shall be tested in radial tension (surface of failure radial) and the other in tangential tension (surface of failure tangential).

Specific Gravity and Shrinkage in Volume

36. Six specific gravity and shrinkage-in-volume specimens shall be selected from the unused portion of bending or tension-parallel-to-grain sticks, selected so as to give one from near the pith, one from near the periphery, and four that are representative of the average growth of the cross-section of the bolt. These specimens shall be selected only from the sticks to be tested in a green condition.

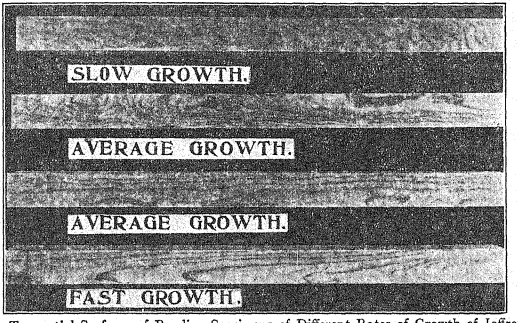


FIG. 4.—Tangential Surfaces of Bending Specimens of Different Rates of Growth of Jeffrey Pine (2 by 2 by 30-in. Specimens).

Cleavage Perpendicular to Grain

34. Eight cleavage specimens shall be selected from four sticks in a manner similar to that for shear (Section 32). One of each pair of specimens from the same stick shall be tested in radial cleavage (surface of failure radial) and the other in tangential cleavage (surface of failure tangential).

Tension Parallel to Grain

35. Four tension-parallel-to-grain specimens shall be chosen of which one shall be selected from near the pith, one from near the periphery, and two which are representative of the cross-section.

Radial Shrinkage

37. Two radial shrinkage specimens shall be obtained from each *d* bolt. They shall be cut from the "sectors" or "quadrants" remaining after sawing (Fig. 2) or from disks cut from near the end of the bolt. When a disk is used, care must be taken to see that it is green, and has not been affected by shrinking and checking which is common near the end of the bolt. The specimens shall not be surfaced. Radial shrinkage specimens shall be cut with their width in the radial direction. One shall be taken from the heartwood, and the other from

near the periphery. When possible, the second shall consist entirely of sapwood.

Tangential Shrinkage

38. Two tangential shrinkage specimens shall be obtained from each *d* bolt. They shall be selected at the same time and in a manner similar to radial-shrinkage specimens (Section 37), except the width shall be in a tangential direction. The specimens shall not be surfaced. One shall be taken from the heartwood, the other from near the periphery and when possible shall consist entirely of sapwood. The heartwood and the sapwood specimens shall be taken adjacent to the respective specimens selected for radial shrinkage.

Photographs of Sticks

Sticks to be Photographed

39. Four of the bending sticks from each species shall be selected for photographing, as follows: two average growth, one fast growth, and one slow growth. These sticks shall be photographed in cross-section and on the radial and tangential surfaces. Fig. 3 is a typical photograph of the cross-section of test specimens and Fig. 4 of the tangential surface of bending specimens.

Control of Moisture Content

Control of Moisture Content

40. As previously specified (Section 22) sticks for test in the air-dry condition shall be brought to practically constant weight before test. Tests shall then be carried out in such manner that large changes in moisture content will not occur. (For instance, in ordinary heated rooms the relative humidity is usually below 30 per cent. If wood which has been dried to practically constant weight under the condition specified by Section 22 is brought into such a room, a considerable and rather rapid drying out will occur.) To prevent such changes, the testing room and rooms for the preparation of test specimens should preferably have some means of humidity control.

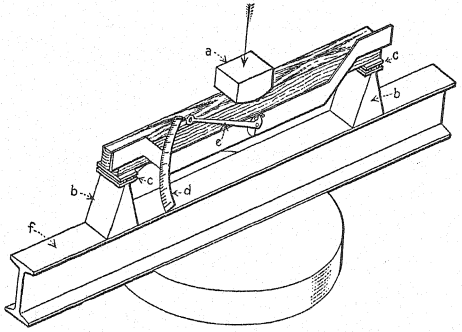


FIG. 5.—Diagrammatic Sketch of Method of Conducting Static Bending Test.

Record of Heartwood and Sapwood

Proportion of Sapwood

41. The estimated proportion of sapwood present shall be recorded for each test specimen.

STATIC BENDING

Size of Specimens

42. The static bending tests shall be made on nominal 2 by 2 by 30-in. specimens. The actual height and width at the center and the length shall be measured (Section 113).

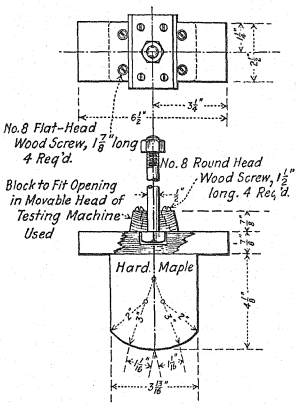


FIG. 6.—Details of Bearing Block for Bending Tests.

Loading Span and Supports

43. Center loading and a span length of 28 in. shall be used. Both supporting knife edges shall be provided with bearing plates and rollers having a total thickness of approximately 1¼ in. in order to bring the neutral plane of the test specimen about 2¼ in. from the knife edges (Fig. 5.)

Bearing Block

44. A bearing block of the form and size of that shown in Fig. 6 shall be used for applying the load.

Placement of Growth Rings

45. The specimen shall be placed so that the load will be applied through the bearing block to the tangential (flat-sawed) surface nearest the pith.

Speed of Testing

46. The load shall be applied continuously throughout the test at a rate of 0.10 in. per min. (Section 114).

Load-Deflection Curves

47. (*a*) Load-deflection curves shall be taken to or beyond the maximum load for all static bending tests. In one-third of the tests, the curves shall be continued to a 6-in. deflection, or until the specimen fails to support a load of 200 lb.

(*b*) Deflections of the neutral plane at the center of the length shall be taken with respect to points in the neutral plane above the supports.

(*c*) Within the elastic limit, deflection readings shall be taken to 0.0025 in. After the elastic limit has been reached, less refinement is necessary in observing deflections, and these may then be read directly to the nearest 0.01 in. by means of a wire stretched across a scale. Figure 5 illustrates the method of test and type of deflectometer recommended for deflections up to 1 in.

(*d*) The load and deflection of first failure, the maximum load, and points of sudden change shall be shown on the curve sheet[7] although they may not occur at one of the regular load or deflection increments.

[7] See Fig. 1 of the Appendix for a sample static bending data sheet form. Fig. 2 of the Appendix shows a sample computation data card.

Description of Failures

48. (*a*) Bending failures shall be classified according to the appearance of the fractured surface and according to the manner in which the failure develops. The fractured surfaces may be roughly divided into brash and fibrous, the term "brash" indicating an abrupt failure and the term "fibrous" indicating a fracture showing splinters. The test failures shall be classified and described as follows:

Simple tension (see Fig. 7*a*),
Cross grain[8] tension (see Fig. 7*b*),

The presence of cross grain having a slope which deviates more than 1 in 20 from the longitudinal edges of the specimen shall be cause for culling the test.

Splintering tension (see Fig. 7*c*),
Brash tension (see Fig. 7*d*),
Compression (see Fig. 7*e*), and
Horizontal shear (see Fig. 7*f*).

(*b*) In case two or more kinds of failures develop, all shall be described in the order of their occurrence; thus, compression followed by splintering tension. The failure shall be sketched on the data sheet.

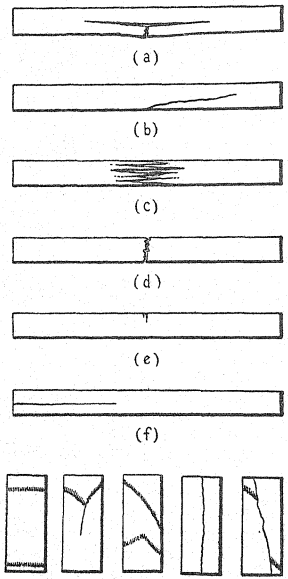


FIG. 7.—Types of Failures: *a* to *f*, Bending; *g* to *k*, Compression Parallel to Grain.

Weight and Moisture Content

49. The specimen shall be weighed after test and a moisture section 1 in. in length shall be cut near the failure (Section 112).

COMPRESSION PARALLEL TO GRAIN

Size of Specimens

50. The compression-parallel-to-grain tests shall be made either (1) on nominal 2 by 2 by 8-in. specimens or (2) on specimens of the special form shown in Fig. 8. The actual cross-section dimensions, or the diameter at minimum section, and the length shall be measured (Section 113).

Advantage of Special Form

51. The specimen with reduced cross-section (Fig. 8) will usually require less care preparatory to test in order to prevent crushing at the ends than the

[8] The term "cross grain" shall be considered to include all deviations of grain from the direction of the longitudinal axis or longitudinal edges of the specimen. It should be noted that spiral grain may be present even to a serious extent without being evident from a casual observation.

specimen with uniform cross-section throughout the length.

End Surfaces Parallel

52. Care shall be used in preparing the compression-parallel-to-grain test specimens to make the end surfaces parallel.

Spherical Bearing

53. A spherical bearing block shall be used and so adjusted for each test as to obtain a uniform distribution of load over the ends of the specimen.

Speed of Testing

54. The load shall be applied continuously throughout the test at a rate of 0.024 in. per min. (Section 114).

Position of Test Failures

56. In order to obtain satisfactory and uniform results, it is necessary that the failures be made to develop in the body of the specimen. With specimens of uniform cross-section, this result can best be obtained when the ends are at a very slightly lower moisture content than the body. With green material it will usually suffice to close-pile the specimens, cover the body with a damp or wet cloth, and expose the ends for a short time. For air-dry material, it may sometimes be advisable to pile the specimens in a similar manner and place them in a desiccator should the failures in test indicate that a slight end drying is necessary.

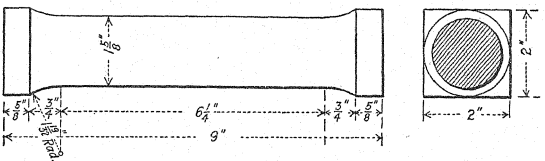


FIG. 8.—Compression-Parallel-to-Grain Test Specimen Special Form.

Load-Compression Curves

55. (*a*) Load-compression curves shall be taken for a 6-in. central gage length on 20 per cent of the specimens. Load-compression readings shall be continued until the elastic limit is well passed, as indicated by the curve.[9] On the other 80 per cent of the specimens the maximum load only will be obtained.

(*b*) Deformations shall be read to 0.0005 in.

(*c*) Figure 9 illustrates a type of compressometer, using a Johnson dial, which has been found satisfactory for wood testing.

[9] See Fig. 4 of the Appendix for a sample compression-parallel-to-grain data sheet form. Figure 3 of the Appendix shows a sample computation data card.

Description of Failures

57. Compression failures shall be classified and described according to their appearance, as follows:

(*a*) *Crushing.*—This term shall be used when the plane of rupture is approximately horizontal. See Fig. 7(*g*).

(*b*) *Wedge Split.*—The direction of the split, that is whether radial or tangential, should be noted. See Fig. 7(*h*).

(*c*) *Shearing.*—This term shall be used when the plane of rupture makes an acute angle with the axis of the specimen. See Fig. 7 (*i*).

(*d*) *Splitting.*—See Fig. 7(*j*).

(*e*) *Compression and Shearing Parallel to Grain.*—This failure usually occurs

in cross-grained pieces, and should be culled as a compression test. See Fig. 7(*k*). The failure shall be sketched on the data sheet. In addition the location of the failure shall be recorded along with its description.

inch and the proportion of summer wood shall be measured over a representative inch of cross-section. In determining the proportion of summer wood, it is essential that the end surface be prepared so as to permit accurate summer wood

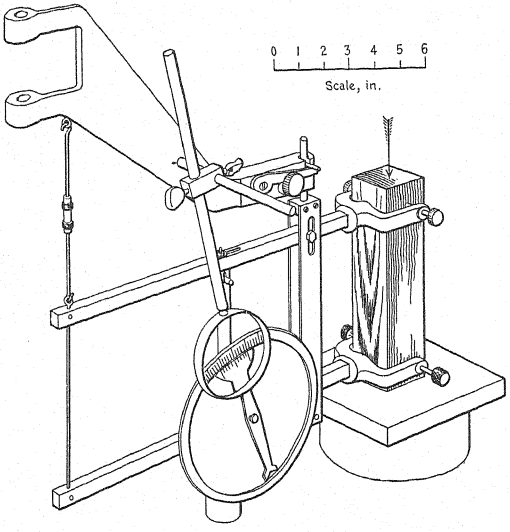


FIG. 9.—Diagrammatic Sketch of U. S. Forest Service Compressometer and Method of Conducting Compression-Parallel-to-Grain Test.

Weight and Moisture Content

58. The specimen shall be weighed after test and a moisture section 1 in. in length shall be cut from the body near the failure (Section 112).

Ring and Summer Wood Measurement

59. After the moisture section has been weighed, the number of rings per

measurement. When the fibers are broomed over at the ends from sawing, a light sanding, planing, or similar treatment of the ends is recommended.

IMPACT BENDING

Size of Specimens

60. The impact bending tests shall be made on nominal 2 by 2 by 30-in.

specimens. The actual height and width at the center and the length shall be measured (Section 113).

Loading and Span

61. Center loading and a span length of 28 in. shall be used.

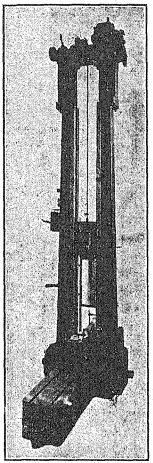


FIG. 10.—Hatt-Turner Impact Machine, Illustrating Method of Conducting Impact Bending Test.

Bearing Block

62. A metal tup of curvature corresponding to the bearing block shown in Fig. 6 shall be used in applying the load.

Placement of Growth Rings

63. The specimen shall be placed so that the load will be applied through the bearing block to the tangential or flat-sawed surface nearest the pith.

Procedure

64. The tests shall be made by increment drops in a Hatt-Turner or similar impact machine (see Fig. 10). The first drop shall be 1 in., which shall be increased by 1-in. increments until a height of 10 in. is reached. A 2-in. increment shall then be used until complete failure occurs or until a 6-in. deflection is reached.

Weight of Hammer

65. A 50-lb. hammer shall be used when, with drops up to the capacity of the machine (about 68 in. for the small Hatt-Turner impact machine), it is practically certain that complete failure or a 6-in. deflection will result for all specimens of a species. For all other cases a 100-lb. hammer shall be used.

Deflection Records

66. Graphical drum records,[10] giving the deflection for each drop and the set, if any, shall be made until first failure occurs. This record shall also afford data from which the exact height of drop can be scaled for at least the first four falls.

Drop Causing Failure

67. The height of drop causing complete failure, or a 6-in. deflection, shall be observed for each specimen.

Description of Failure

68. The failure shall be sketched on the data sheet[11] and described in accordance with the directions for static bending under Section 48.

Weight and Moisture Content

69. The specimen shall be weighed after the test and a moisture section

[10] See Fig. 5 of the Appendix for a sample drum record.
[11] See Fig. 7 of the Appendix for a sample impact-bending data sheet form. Figure 6 of the Appendix shows a sample computation data card.

1 in. in length shall be cut near the failure (Section 112).

COMPRESSION PERPENDICULAR TO GRAIN

Size of Specimens

70. The compression-perpendicular-to-grain tests shall be made on nominal 2 by 2 by 6-in. specimens. The actual height, width, and length shall be measured (Section 113).

Speed of Testing

73. The load shall be applied continuously throughout the test at a rate of 0.024 in. per min. (Section 114).

Load-Compression Curves

74. (*a*) Load-compression curves[12] shall be taken for all specimens up to 0.1-in. compression, after which the test will be discontinued.

(*b*) Deflection readings shall be taken to 0.001 in.

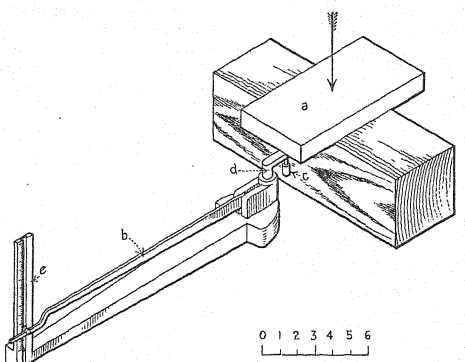


FIG. 11.—Diagrammatic Sketch of Method of Conducting Compression-Perpendicular-to-Grain Test.

Loading

71. The load shall be applied through a metal bearing plate 2 in. in width, placed across the upper surface of the specimen at equal distances from the ends and at right angles to the length. (See Fig. 11.)

Placement of Growth Rings

72. The specimen shall be placed so that the load will be applied through the bearing plate to a radial (quarter-sawed) surface.

Weight and Moisture Content

75. The specimen shall be weighed after test and a moisture section 1 in. in length shall be cut adjacent to the part under load (Section 112).

HARDNESS

Size of Specimens

76. The hardness tests shall be made on nominal 2 by 2 by 6-in. specimens.

[12] See Fig. 8 of the Appendix for a sample compression-perpendicular-to-grain data sheet form. Figure 9 of the Appendix shows a sample computation data card.

The actual cross-section dimensions and length shall be measured (Section 113).

Procedure

77. The modified ball test with a "ball" 0.444 in. in diameter shall be used for determining hardness. (See Fig. 12.) The load at which the "ball" has penetrated to one-half its diameter as indicated by the tightening of the collar against the specimen shall be observed.

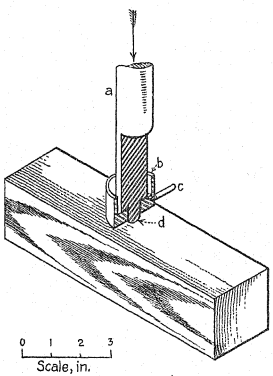


FIG. 12.—Diagrammatic Sketch of Method of Conducting Hardness Test.

Number of Penetrations

78. Two penetrations shall be made on a tangential surface, two on a radial surface, and two on each end. The choice between the two radial and between the two tangential surfaces shall be such as to give a fair average of the piece. The penetrations shall be far enough from the edge to prevent splitting or chipping.[13]

Speed of Testing

79. The load shall be applied continuously throughout the test at a rate of 0.25 in. per min. (Section 114).

Weight and Moisture Content

80. The specimen shall be weighed after test and a moisture section 1 in. in length shall be cut (Section 112).

SHEAR PARALLEL TO GRAIN

Size of Specimens

81. The shear-parallel-to-grain tests shall be made on nominal 2 by 2 by $2\frac{1}{2}$-in. specimens notched as illustrated in Fig. 13 to produce failure on a 2 by 2-in. surface. The actual dimensions of the shearing surface shall be measured (Section 113).

Procedure

82. A shear tool similar to that illustrated in Fig. 13, providing a $\frac{1}{8}$-in. offset between the inner edge of the supporting surface and the plane along which the failure occurs, shall be used. The load shall be applied and the specimen supported on end-grain surfaces. Care shall be taken in placing the specimen in the shear tool to see that the cross bar is adjusted so that the edges of the specimen are vertical and the end rests evenly on the support over the contact area. The maximum load only will be observed.

Speed of Testing

83. The load shall be applied continuously throughout the test at a rate of 0.015 in. per min. (Section 114).

Test Failures

84. The failure shall be sketched on the data card.[14] In all cases where the failure at the base of the specimen ex-

[13] See Fig. 10 of the Appendix for a sample data and computation card for hardness test.

[14] See Fig. 11 of the Appendix for a sample tangential-shear-parallel-to-grain data sheet form.

tends back onto the supporting surface, the test shall be culled.

Moisture Content

85. The portion of the test piece which is sheared off shall be used as a moisture specimen (Section 112).

TENSION PERPENDICULAR TO GRAIN

Size of Specimens

86. The tension-perpendicular-to-grain tests shall be made on specimens

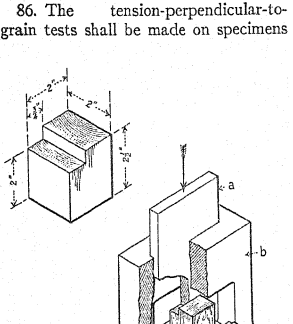


FIG. 13.—Diagrammatic Sketch of Method of Conducting Shear-Parallel-to-Grain Test, with Details of Test Specimen.

of the nominal size and shape shown in Fig. 14. The actual width and length at minimum sections shall be measured (Section 113).

Procedure

87. The specimens shall be held during test in grips as shown in Fig. 14. The maximum load only shall be observed.

Speed of Testing

88. The load shall be applied continuously throughout the test at a rate of 0.25 in. per min. (Section 114).

Sketch of Failure

89. The failure shall be sketched on the data card.[15]

Moisture Content

90. One of the pieces remaining after failure, or a section split along the surface of failure, shall be used as a moisture specimen (Section 112).

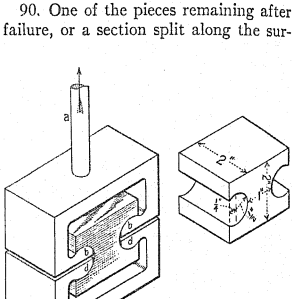


FIG. 14.—Diagrammatic Sketch of Method of Conducting Tension-Perpendicular-to-Grain Test, with Details of Test Specimen.

CLEAVAGE

Size of Specimens

91. The cleavage tests shall be made on specimens of the form and size shown in Fig. 15. The actual width and length at minimum section shall be measured (Section 113).

[15] See Fig. 12 of the Appendix for a sample radial-tension-perpendicular-to-grain data sheet form.

Procedure

92. The specimens shall be held during test in grips as shown in Fig. 15. The maximum load only shall be observed.

Speed of Testing

93. The load shall be applied continuously throughout the test at a rate of 0.25 in. per min. (Section 114).

Sketch of Failure

94. The failure shall be sketched on the data card.[16]

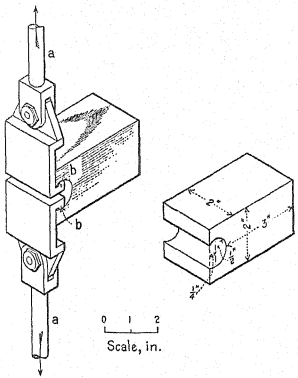


FIG. 15.—Diagrammatic Sketch of Method of Conducting Cleavage Test, with Details of Test Specimen.

Moisture Content

95. One of the pieces remaining after failure, or a section split along the surface of failure, shall be used as a moisture specimen (Section 112).

TENSION PARALLEL TO GRAIN

Size of Specimens

96. The tension-parallel-to-grain tests shall be made on specimens of the size and shape shown in Fig. 16. The actual cross-section dimensions at minimum section shall be measured (Section 113).

Procedure

97. The specimen shall be held during test by plates notched to fit around the test piece and under the 0.25-in. shoulder provided for that purpose. A spherical bearing shall be used on at least one end of the specimen. Maximum load only will be observed.

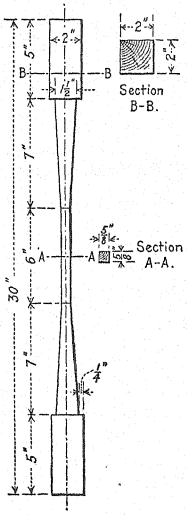


FIG. 16.—Details of Tension-Parallel-to-Grain Test Specimen.

Speed of Testing

98. The load shall be applied continuously throughout the test at a rate of 0.05 in. per min. (Section 114).

[16] See Fig. 13 of the Appendix for a sample radial-cleavage data sheet form.

Sketch of Failure

99. The failure shall be sketched on the data sheet.[17]

Moisture Content

100. A moisture section about 3 in. in length shall be cut from the minimum section near the failure (Section 112).

actual cross-section dimensions and length shall be measured (Section 113).

Procedure

102. (*a*) Both specific gravity and shrinkage-in-volume determinations shall be obtained on the same specimen.

(*b*) A carbon impression of the end of

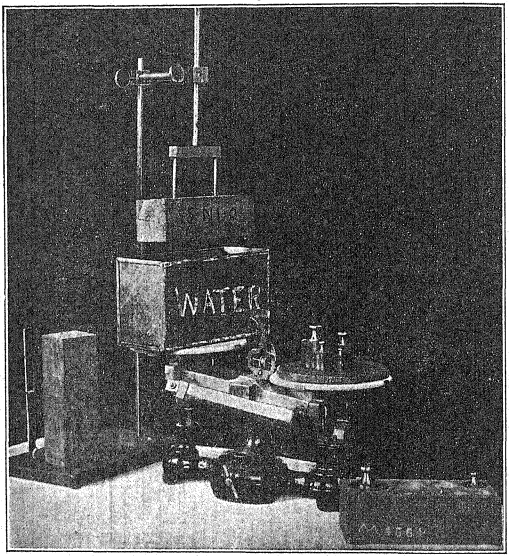


FIG. 17.—Method of Conducting Specific Gravity and Shrinkage-in-Volume Test.

SPECIFIC GRAVITY AND SHRINKAGE IN VOLUME

Size of Specimens

101. The specific gravity and shrinkage in volume tests shall be made on nominal 2 by 2 by 6-in. specimens. The

the green specimen shall be made on the back of the data card.[18] In like manner, a carbon impression of the same end shall be made after the specimen has been oven dried. (See Paragraph (*e*).)

(*c*) The specimen shall be weighed when green (Section 112) and the volume

[17] See Fig. 14 of the Appendix for a sample tension-parallel-to-grain data sheet form.

[18] See Fig. 15 of the Appendix for a sample specific gravity and shrinkage-in-volume data card form.

shall be determined by the immersion method.

(*d*) The green specimens after immersion shall be open-piled and allowed to air-season under room conditions until approximately constant weight is attained.

(*e*) The specimens shall then be open-piled in an oven and dried at 100 C. until constant weight is reached.

(*f*) After oven drying, the specimens shall be weighed (Section 112) and while still warm shall be immersed in a hot paraffin bath, care being taken to remove them quickly to insure a thin coating.

(*g*) The volume of the paraffin-coated specimen shall be determined by immersion as before.

(*h*) Figure 17 illustrates the apparatus used in determining the specific gravity and shrinkage in volume.

RADIAL AND TANGENTIAL SHRINKAGE

Size of Specimen

103. The radial and tangential-shrinkage determinations shall be made on nominal 1 by 4 by 1-in. specimens. The specimen shall be measured across the (4-in.) dimension in which the shrinkage is to be determined (Section 113).

Weight

104. The specimen shall be weighed when green and after subsequent oven drying (Section 112).

Drying

105. (*a*) The green specimens shall be open-piled and allowed to air-season under room conditions until approximately constant weight is attained.

(*b*) The specimens shall then be open-piled in an oven and dried at 100 C. until constant weight is attained.

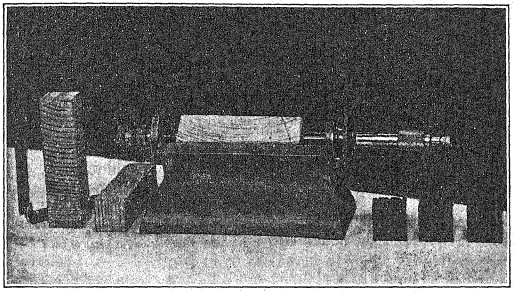


FIG. 18.—Method of Measuring Radial and Tangential Shrinkage.

Final Measurement

106. Final measurement shall be made on the oven-dry specimen.[19]

Method of Measurement

107. Figure 18 illustrates the method of making the radial and tangential-shrinkage measurements. An ordinary micrometer of required accuracy is suitable for this work (Section 113).

MOISTURE DETERMINATION

Selection

108. The sample for moisture determinations of each test specimen shall be

[19] See Fig. 16 of the Appendix for a sample radial and tangential-shrinkage data card form.

selected as heretofore described for each test.

Weighing

109. Immediately after obtaining the moisture sample, all loose splinters shall be removed and the sample shall be weighed (Section 112).

Drying

110. The moisture samples shall be open-piled in an oven and dried at a temperature of 100 C. until constant weight is attained, after which the oven-dry weight shall be determined (Section 112).

Moisture Content

111. The loss in weight expressed in percentage of the oven-dry weight as above determined shall be considered the moisture content of the specimen.

PERMISSIBLE VARIATIONS

Weights

112. The weight of test specimens and of moisture samples shall be determined to an accuracy of not less than plus or minus 0.2 per cent.

Measurements

113. Measurements of test specimens shall be made to the nearest 0.01 in. except in the case of tension parallel to grain, radial shrinkage, and tangential shrinkage, when measurements shall be taken to the nearest 0.001 in.

Testing Machine Speeds

114. In no case shall the testing machine speed used vary more than 25 per cent from that specified for a given test. The load shall be applied continuously, without interruption, at the required speed throughout the test.

CALIBRATION

Calibration

115. All apparatus used in obtaining data shall be calibrated at sufficiently frequent intervals to insure accuracy.[20]

[20] Standard Methods of Verification of Testing Machines (A.S.T.M. Designation E 4), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

APPENDIX

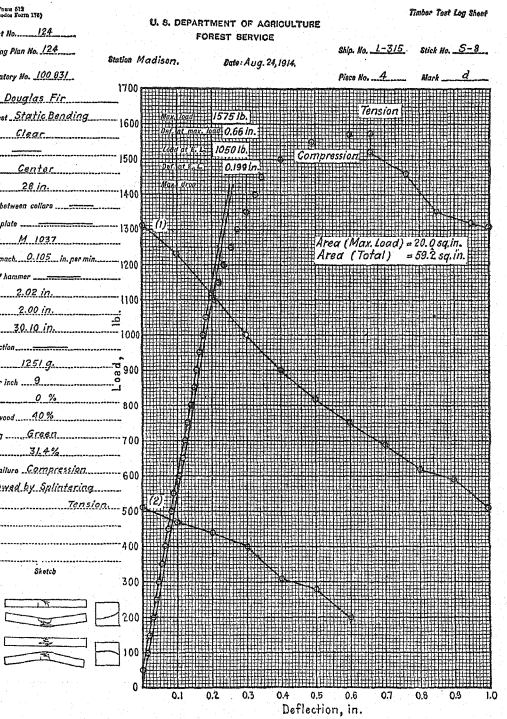


FIG. 1.—Sample Data Sheet for Static Bending Test.

STATIC BENDING

1-315 (Shipp. No.) S-8 (Piece No.) Center Loading 100 831 (Lab. No.)

4 (Piece No.) d (Mark) Station MADISON Date Aug. 24-'14 124 (Project No.)

Species Douglas Fir Grade Clear Seasoning Green

Rings 9 Sap 0 % Summerwood 40 % Moisture 31.4 %

Span 28 in. Length 30.10 in. Height 2.02 in. Width 2.00 in. Weight 1251 g.

SPECIFIC GRAVITY, AS TEST.	SPECIFIC GRAVITY, OV. DRY.	F. S. AT E. L.	H. OF R.	M. OF E.	SHEAR.	WORK TO E. L.	WORK TO MAX. LOAD.	TOTAL WORK.	
0.628	0.478	5410	8120	1756	292	0.92	7.1	20.9	

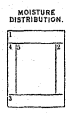
Rings: Up. ¼ Mid. ¼ Low. ¼

Sum. wood: Up. ¼ Mid. ¼ Low. ¼

Defects

Failure Compression Followed by Splintering Tension

MOISTURE DISTRIBUTION.



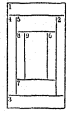


FIG. 2.—Sample Computation Data Card for Static Bending Test.

COMPRESSION PARALLEL TO GRAIN

1-315 (Shipp. No.) E-11 (Block No.) 101 101 (Lab. No.)

1 (Piece No.) d-1 (Mark) Station Madison Date Aug. 25, 1914 124 (Project No.)

Species Douglas Fir Grade Clear Seasoning Green

Rings 9 Sap 90 % Summerwood 30 % Moisture 57.6 %

Length 8.00 in. Cross section 2.01 in. X 2.01 in. Weight 365 g.

SPECIFIC GRAVITY, AT TEST	SPECIFIC GRAVITY, OV. DRY	MAX. LOAD	CRUSH. ST. AT E. L.	MAX. CRUSH. ST.	M. OF E.	LOAD AT E.L.	DEF. AT E.L.
0.690	0.438	14 760	3220	3660	1932	13 000	0.0097

Defects

Failure Shear at Top

FIG. 3.—Sample Computation Data Card for Compression-Parallel-to-Grain Test.

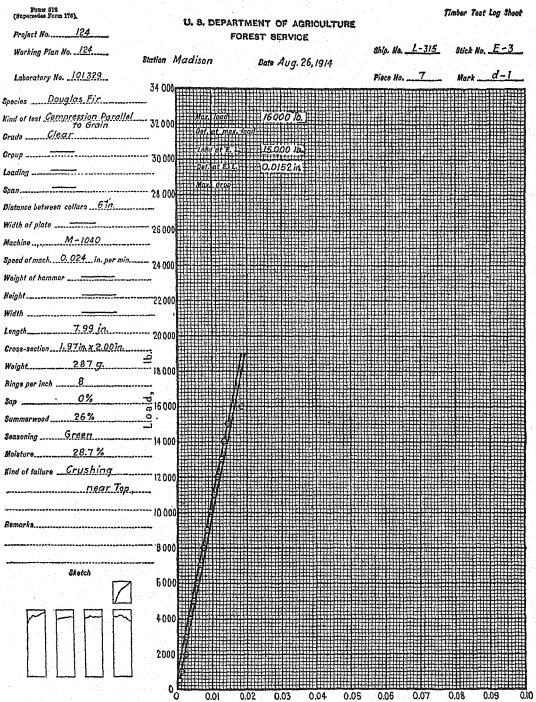


FIG. 4.—Sample Data Sheet for Compression-Parallel-to-Grain Test.

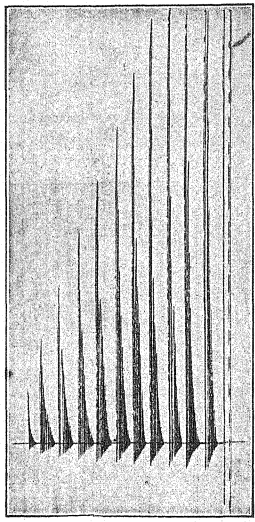


FIG. 5.—Sample Drum Record of Impact Test.

IMPACT BENDING

L-315 (Bldg. No.) | E-12 (Stock No.) | 101 151 (Lab. No.)

1 (Piece No.) | C (Mark) | Station MADISON | Date Aug. 20, 1914 | 124 (Project No.)

Species Douglas Fir | Grade Clear | Seasoning Green

Rings 8 | Sap 100 % | Summerwood 30 % | Moisture 61.4 %

Hammer 50 lbs. | Span 28 in. | Length 29.94 in. | Height 2.00 in. | Width 2.00 in. | Weight 1370 g.

Drop No.	Head.	Def.	$\frac{W}{b^2}$	Set.	Drop No.	Head.	Def.	$\frac{Def}{l^2}$	Set.
1	1.0	0.13	0.017		11	12.0	0.50	0.250	
2	2.0	0.18	0.032		12	14.0	0.55	0.302	
3	3.0	0.22	0.048		13	16.0	0.62	0.384	
4	4.0	0.26	0.068		14	18.0	0.67	0.593	
5	5.0	0.30	0.090		15				
6	6.0	0.34	0.116		16				
7	7.0	0.36	0.130		17				
8	8.0	0.38	0.144		18				
9	9.0	0.43	0.185		19				
10	10.0	0.46	0.212		20				

Sp. Gr. (at test),	0.698
Sp. Gr. (oven dry),	0.432
$P.S.$ at E. L.,	10 610
$M.$ of R.,	1776
E. Resil.,	3.51
Max. Drop,	22 in.
s_1	0.010
H	7.88
Δ	0.39

Failure: Compression, Followed by Splintering Tension.

FIG. 6.—Sample Computation Data Card for Impact-Bending Test.

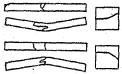
U. S. DEPARTMENT OF AGRICULTURE
FOREST SERVICE

Project No. 124
Working Plan No. 124
Station Madison
Date Aug. 26, 1914.
Ship. No. L-315
Stock No. F-12
Laboratory No. 101.151
Piece No. 1
Mark C

Species Douglas Fir
Kind of test Impact Bending
Grade Clear
Group
Loading Center
Span 28 in.
Distance between collars
Width of plate
Machine Hatt-Turner
Speed of mach. in. per min.
Weight of hammer 50 lb.
Height 2.00 in.
Width 2.00 in.
Length 29.94 in.
Cross-section
Weight 1370 g.
Rings per inch 8
Sap 100%
Summerwood 30 %
Seasoning Green
Moisture 61.4%
Kind of failure Compression followed by Splintering Tension

Remarks

Sketch



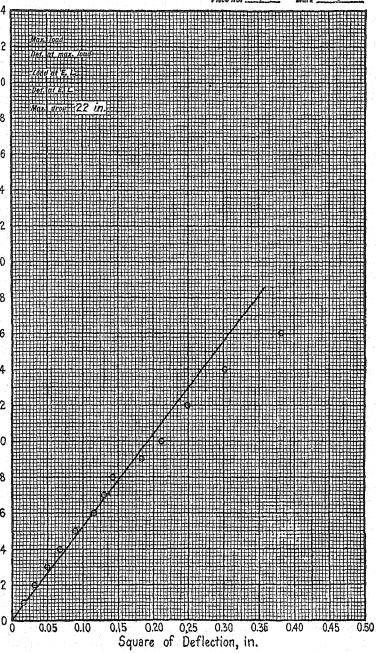


FIG. 7.—Sample Data Sheet for Impact-Bending Test.

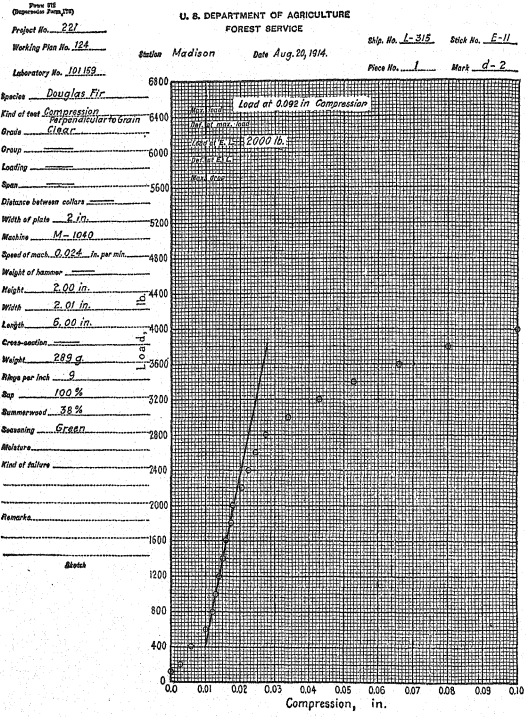


FIG. 8.—Sample Data Sheet for Compression-Perpendicular-to-Grain Test.

L-315 (Shlp. No.) E-11 (Stick No.) COMPRESSION PERPENDICULAR TO GRAIN 101159 (Lab. No.)

1 (Piece No.) d-2 (Mark) Station MADISON Date Aug. 20, 1914 124 (Project No.)

Species Douglas Fir Grade Clear Seasoning Green

Rings 9 Sap 100 % Summerwood 38 % Moisture 62.1 %

Width of plate 2 in. Length 6.00 in. Height 2.00 in. Width 2.01 in. Weight 289 g.

Specific Gravity		Load at E. L.	Crush. St. at E. L.	$\Delta \div h$	
At Test.	Ov. Dry				
0.732	0.452	2000	498		

8—1656

FIG. 9.—Sample Computation Data Card for Compression-Perpendicular-to-Grain Test.

HARDNESS

L-315 (Shlp. No.) E-10 (Stick No.) 101170 (Lab. No.)

1 (Piece No.) d-4 (Mark.) Station MADISON Date Aug. 20, 14 124 (Project No.)

Species Douglas Fir Grade Clear Seasoning Green

Rings 8 Sap — % Summerwood 33 % Moisture 31.7 %

Length 6.01 in. Cross section 2.00 in. x 2.00 in. Weight 246 g.

	Specific Gravity.		Radial Surface.	Tangential Surface.	End Surface.
	At Test.	Ov. Dry.			
1	0.622	0.472	460	570	525
2			520	460	500
3					510
4					510
Avg.,			490	515	511
Avg. Rad. and Tang.,			502		

Sketch.

8—1422

FIG. 10.—Sample Data and Computation Card for Hardness Test.

Tangential SHEAR

L-315 (Ship. No.) E-12 (Stick No.) 101 189 (Lab. No.)

1 (Piece No.) C-5 (Mark) Station MADISON Date Aug. 21-14 124 (Project No.)

Species Douglas Fir Grade Clear Seasoning Green

Rings 8 Sap 75 % Summerwood 30 % Moisture 800 %

Shearing Area	Max. Load	Shearing Str.	Time		Sketch
2.02 x 2.01	3600	887			→

8–1455

FIG. 11.—Sample Data and Computation Card for Tangential-Shear-Parallel-to-Grain Test. (Surface of failure tangential.)

R TENSION PERPENDICULAR TO GRAIN

L-315 (Ship. No.) E-12 (Stick No.) 101 189 (Lab. No.)

1 (Piece No.) C-6 (Mark) Station MADISON Date Aug. 21-14 124 (Project No.)

Species Douglas Fir Grade Clear Seasoning Green

Rings 8 Sap 80 % Summerwood 30 % Moisture 44.2 %

Height.	Width.	Length.	Maximum Load.	Tension, lbs. per sq. in.	Sketch.
	2.01	0.97	533	273	

8–2515

FIG. 12.—Sample Data and Computation Card for Radial-Tension-Perpendicular-to-Grain Test (Surface of failure radial.)

P CLEAVAGE

L-315 (Ship. No.) E-12 (Stick No.) 191171 (Lab. No.)

1 (Piece No.) C-5 (Mark) Station MADISON Date Aug. 21.-14. 124 (Project No.)

Species Douglas Fir Grade Clear Seasoning Green

Rings 8 Sap 75 % Summerwood 30 % Moisture 31.6 %

HEIGHT.	WIDTH.	LENGTH.	MAX. LOAD.	LOAD PER INCH WIDTH.
	2.01	2.98	365	182

SKETCH.

8—1433

FIG. 13.—Sample Data and Computation Card for Radial-Cleavage Test. (Surface of failure radial.)

TENSION PARALLEL TO GRAIN

606 (Ship. No.) HM-14 (Stick No.) 347416 (Lab. No.)

2 (Piece No.) 5-F (Mark) Station MADISON Date Jan. 23, '19 221-1 (Project No.)

Species Douglas Fir Grade Clear Seasoning Green

Rings 10 Sap — % Summerwood 31 % Moisture 32.5 %

CROSS SECTION.	LENGTH.	MAXIMUM LOAD.	TENSION, Lbs. per sq. in.
0.641 X 0.617		4670	11810

8—2515

FIG. 14.—Sample Data and Computation Card for Tension-Parallel-to-Grain Test.

SPECIFIC GRAVITY AND VOLUMETRIC SHRINKAGE

L-315 (SHIP. NO.) S-6 (STICK NO.) STATION, Madison, Aug. 20,-14 (DATE) 101197 (LAB. NO.)

1 (PIECE NO.) d-8 (MARK) 124 (PROJECT NO.)

SPECIES Douglas Fir

NOMINAL SIZE OF SPECIMEN 2 in. x 2 in. x 6 in. % SAP 0 % SUMMER WOOD 40

	DATE	RINGS PER INCH	WEIGHT, GRAMS	% MOIST	VOLUME C. C.	SPECIFIC GRAVITY	WEIGHT, LBS. PER CU. FT.	X % VOL. SHRINKAGE
GREEN	Aug. 20,-14	8	253	33.2	398	0.636	39.6	13.8
AIR DRY								
KILN DRY								
OVEN DRY	Sep. 25,-14		190		343	0.554	34.5	

X BASED ON ORIGINAL VOLUME (GREEN, AIR DRY, KILN DRY) 1ST WT. .....

NOTE—USE BACK OF CARD FOR CARBON IMPRESSIONS 2D WT. .....

REMARKS: ..... VOL. .....

FIG. 15.—Sample Data and Computation Card for Specific Gravity and Shrinkage-in-Volume Test.

SHRINKAGE—RADIAL AND TANGENTIAL

L-315 (SHIP. NO.) (STICK NO.) STATION—MADISON, WISCONSIN 101200 101199 (LAB. NOS.)

1 (PIECE NO.) d (MARK) 124 (PROJECT NO.)

SPECIES Douglas Fir

NOMINAL SIZE OF SPECIMEN 1 in. X 4 in. x 1 in.

SEASONING	DATE	RINGS PER INCH	% SAP	% SUMMERWOOD	WIDTH INCHES	WEIGHT GRAMS	% MOISTURE	X % SHRINKAGE
RADIAL								
GREEN	Aug. 19-14	11	30	41	4.006	49.8	66.5	
OVEN-DRY	Oct. 5-14				3.808	29.9		4.9
TANGENTIAL								
GREEN	Aug. 19-14	12	95	34	4.016	64.0	119.1	
OVEN-DRY	Oct. 5-14				3.632	29.2		9.5

X BASED ON GREEN WIDTH

FIG. 16.—Sample Data and Computation Card for Radial and Tangential-Shrinkage Tests.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: O4b-1927

Standard Methods of

STATIC TESTS OF TIMBERS IN STRUCTURAL SIZES[1]

A.S.T.M. Designation: D 198 - 27

ADOPTED, 1927.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 198; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Although numerous tests of timber in structural sizes have been made for various purposes, there has been a general lack of uniformity of test procedure. Consequently, it has not been possible to correlate data from different sources, nor have the results obtained by different investigators contributed their full measure of usefulness toward an increasing fund of knowledge. Study and experience have shown the extent to which various factors, such as loading appliances, rate of loading, etc., influence test results, and it is with the thought of standardizing these and other factors that this test procedure is prepared. The plan has been to cover very definitely the tests of certain types of timbers such as bridge stringers and joists and at the same time to suggest procedure, so far as possible, for other structural timber forms. Summarized, the underlying purpose is the establishment of practice which will afford uniform results of the greatest ultimate value from all tests of timbers and at the same time permit the correlation of data so far as possible with existing data on timbers of certain species.

It is not the intention to discourage the use of methods other than those herein prescribed whenever the testing problem is of such nature as to warrant their application. However, experience has emphasized the importance of the requirements that have been specified in these methods, and variations in apparently minor details will often seriously affect the data obtained. It is recommended that any variations from the methods herein prescribed shall be recorded.

The field of timber testing is so broad, and involves so many objectives, particularly when built-up or manufactured articles are considered, that it is obviously difficult, if not impossible, to formulate correct procedure for all such cases. Ample opportunity, therefore, still awaits the application of initiative to special testing problems and promotion of progress in the art. It is hoped, however, that the principles incorporated in these methods will at least serve to some extent as a guide in tests of even a special nature.

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-7 on Timber

[2] Prior to adoption as standard, these methods were published as tentative from 1924 to 1927.

The test methods presented are the outgrowth of years of experience and research, largely on the part of the United States Forest Service. Such sample data forms are included as were thought helpful to investigators in systematizing records. No attempt has been made to cover methods of computation or analysis, as these were considered to lie outside the scope of the standard. Data properly recorded can be analyzed at will, but fundamental differences in test procedure present a barrier beyond which analysis and correlation of results cannot proceed with any degree of assurance.

Purpose of Tests

1. Tests of timbers in structural sizes are made to afford:

(*a*) Data upon which to base the formulation of grading rules and specifications.

(*b*) Data upon which to establish the relation between the strength properties of structural timbers and of small clear specimens cut from them, and for use in the establishment of allowable stresses.

(*c*) Data as to the influence of defects on the mechanical properties of timbers.

(*d*) Data on the strength properties of different species in structural sizes.

(*e*) Data for use in checking existing formulas relating to structural timbers.

(*f*) Data as to the influence of seasoning on the mechanical properties.

(*g*) Data as to the effect of form and shape of specimen on the properties.

(*h*) Data as to the influence of preservatives and methods of preservative treatment on the mechanical properties.

Classes of Tests

2. In general, tests of timbers are divided into two classes, as follows:

(*a*) Major tests, that is, tests of principal members under investigation.

(*b*) Minor tests, that is, tests of small clear specimens cut from the principal members.

Kind of Tests

3. (*a*) The principal tests of such structural timbers as bridge stringers or joists are:

Static bending,
Compression parallel to grain, and
Compression perpendicular to grain.

(*b*) The accompanying minor tests on small clear specimens are:

Static bending,
Compression parallel to grain,
Compression perpendicular to grain,
Hardness, and
Shear parallel to grain.

COLLECTION OF MATERIAL

Selection

Authentic Identification

4. The material for test shall be selected by one qualified to identify the species.

Procedure for Bridge Stringers, Joists, etc.

5. In any given case, the number of test specimens shall be determined from due consideration of the purpose in mind, since definite instructions cannot be given to cover all cases. The following procedure applies particularly to full-size timbers, such as bridge stringers and joists, and incidentally to other types, as fabricated beams, to which special reference will be made in paragraphs set in smaller type.

Number and Size of Major Specimens

6. (*a*) For tests of timbers of any given species in structural sizes, such as bridge stringers, requiring a range in grade of material, at least forty 32-ft. or eighty 16-ft. green unsurfaced timbers should be selected in a manner that will provide representative material. The length of specimens required for test is

dependent upon the depth. For suggested lengths of timbers other than bridge stringers, see Section 22 (*b*), (*c*), and (*d*).

(*b*) Timbers 8 by 16 in. or 6 by 12 in. in nominal cross-section are especially recommended.

Selection and Matching of Specimens

7. Each 32-ft. timber shall be cut in half to furnish one 16-ft. specimen to be tested green and one to be tested air-dry, thus affording end matching. Where only 16-ft. material is available, the timbers shall be selected and matched in pairs, one timber to be tested green and the other to be tested air-dry. In matching these 16-ft. timbers all factors influencing their strength should, so far as practicable, be considered. Some of the most important factors are density, rate of growth, direction of grain (straight, diagonal, or spiral), character and location of defects (such as knots, checks, etc.), position of specimen in tree with respect to cross-section or height, and the characteristics of individual trees.

Field Marking

Butt Designation

8. At the time of selection a mark shall be made on each timber to designate the butt end.

Shipment Number

9. All material collected at a given source and shipped at one time shall be given a shipment number or other designation which shall be incorporated in the test records.

Field Descriptions

Field Descriptions

10. Field notes fully describing the material shall be carefully made by the collector. These notes shall, so far as possible, supply data as outlined in Table I and shall be forwarded for incorporation in the record.

TABLE I.—SHIPMENT DESCRIPTION FIELD NOTES.

Project No., *XC* — Shipment No., *012*
Tree No., *1* — Species, *Longleaf pine*
Locality cut: State, *Florida* — County, *Nassau*
Township, *T3N: R25E* — Date cut, *April, 1914*
Slope, *Level.* Absolute elevation, *38 ft.* — Aspect, ...
Undergrowth, *Small amount—scattered* — Soil, *Sandy*
Height, *110 ft.* Seedling or sprout, *Seedling* — Age, *241 years*
Crown, *Large, well developed* — D.B.H., *26 in.*
Stem, *Clear, straight* — Date sawed, *May 12, 1914*
How and when transported from woods, *By wagon, May, 1914*

LIST OF SPECIMENS.

Piece No.	Size of Specimens			Volume, cu. ft.	Footage, b. m.	Weight, lb. (Show date)
	Length, ft.	Width, in.	Depth, in.			
1	16.10	7.50	15.50	13.00	171	660

REMARKS: Note any other special points which would be of value.

Preparation for and Records of Shipment

Preparation for Shipment

11. (*a*) The ends of all green timbers shall be given a coating of paint or other suitable substance to retard end drying and end checking, care being taken to retain the butt designation. They shall be close-piled, and before and during shipment shall be kept covered with damp shavings or other substance to prevent drying. If conditions are particularly favorable to the development of molds, stains, or decay, the specimens shall first be brushed or sprayed with a dilute solution of copper sulfate, or similar toxic solution.

(*b*) Record shall be made of the ship-

ment routing, bill of lading, kind of shipment, date of shipment, and condition of material when shipped.

(*c*) Record shall also be made of date of receipt of shipment at destination, its condition and the method of storage. The time elapsing between the sawing of green timbers and their testing should be reduced to a minimum.

DISPOSITION OF MATERIAL AT DESTINATION

Grouping

12. When the material arrives at the testing laboratory, it shall be divided into two matched groups of timbers of approximately like quality as specified in Section 7, one portion to be tested green and the other to be weighed and placed under cover for air seasoning prior to testing. After the 32-ft. timbers have been cut into 16-ft. lengths, the specimens to be air-dried shall be selected alternately from butt and top cuts.

Marking

13. The butt end of each 16-ft. specimen shall be designated "*A*" and the top end "*B*". Other symbols shall be given to each timber in classifying it as to density, position in tree, defects, or other factors that are under consideration. The minor test specimens and data forms shall carry such of these symbols as may be necessary to identify them fully.

Storage

14. (*a*) The material to be tested green shall be close-piled on skids which are free from contact with the soil, and shall be kept covered. It shall not be held long enough to permit seasoning[3] or damage from checking, decay, or insect attack.

(*b*) The ends of the specimens to be air-dried shall, if necessary, be given a suitable coat of pitch, paint, or other substance to retard checking. The specimens shall be open-piled in a place allowing free access and circulation of air, but shall be protected from sunshine, rain, snow, and moisture from the ground and shall not be subjected to artificial heat.

(*c*) All of the timbers to be air-dried shall be weighed when stored and at sufficiently frequent intervals thereafter to afford accurate data on the progress of seasoning. No material shall be considered thoroughly air-dry and fully conditioned for testing until practically constant weight has been reached.[4] (Wood absorbs and gives off moisture with changing atmospheric conditions; consequently it never comes to absolutely constant weight.)

(*d*) When properly seasoned, the timbers shall be surfaced on four sides before being tested.

KIND AND NUMBER OF TESTS

Specimens for Major Tests[5]

15. (*a*) *Major Bending.*—The structural member proper shall be tested as a beam. (See Sections 19 to 30.)

(*b*) *Compression Perpendicular to Grain.*—A section 60 in. long shall be cut from the "*A*" or butt end of each beam after test, except when end "*A*" fails by shear in the bending test, in

[3] The many factors which influence the rate of seasoning, such as species, moisture content, and diverse climatic differences, preclude the establishment of a definite limit of loss in weight which may be permitted in green material prior to test.

[4] A desirable condition for testing air-day material in small sizes may be considered as about 12 per cent moisture content. Large timbers, such as bridge stringers, however, usually dry slowly, but may be considered conditioned when, during the season of most rapid drying, successive weighings indicate the moisture content is as low as may reasonably be expected, with due consideration to the climatic conditions of the locality. Such seasoning may require from about one to four years.

[5] The procedure outlined utilizes to best advantage the entire major specimen and if carefully followed will not usually admit the inclusion of specimens which have been seriously strained in the major bending test. The compression specimens referred to in Sections 15(*b*) and (*c*) necessarily include such defects as are normal to the material under test.

which case the section shall be taken from the "*B*" or top end. A specimen 30 in. long for compression-perpendicular-to-grain test shall be cut from that end of the 60-in. piece which is nearer the point of loading.

(*c*) *Compression Parallel to Grain.*—Two compression-parallel-to-grain specimens, each 6 by 6 by 24 in., shall be taken from the farther end of the 60-in. piece specified in Paragraph (*b*).

Specimens for Minor Tests[6]

16. (*a*) A 50-in. section shall be cut from the end of the beam opposite that required for the 60-in. specimen specified in Section 15 (*b*). Six 2 by 2 by 50-in. sticks shall be taken from this piece. In case defects make it difficult to obtain certain of the other clear specimens hereinafter provided for, they may be secured from the uninjured portion of the minor static bending specimens after the test covered in Paragraph (*b*).

(*b*) *Static Bending.*—Six static bending specimens free from defects shall be taken, one from each of the 50-in. sticks specified in Paragraph (*a*).

(*c*) *Compression Parallel to Grain.*—Six compression-parallel-to-grain specimens free from defects shall be taken, one from each of the 50-in. sticks specified in Paragraph (*a*).

(*d*) *Compression Perpendicular to Grain.*—Three compression-perpendicular-to-grain specimens free from defects shall be taken, one from each of three of the 50-in. sticks specified in Paragraph (*a*).

(*e*) *Hardness.*—Three hardness specimens free from defects shall be taken, one from each of three of the 50-in. sticks specified in Paragraph (*a*) other than those from which the specimens specified in Paragraph (*d*) are taken.

(*f*) *Shear Parallel to Grain.*—Twelve shear specimens free from defects shall be cut, two from each of the 50-in. sticks specified in Paragraph (*a*). One specimen from each pair taken from the same stick shall be tested in radial shear (surface of failure radial to growth rings) and the other in tangential shear (surface of failure tangential to growth rings).

Photographs of Timbers

17. All air-dried timbers shall be photographed on four sides both before and after test. All green timbers shall be photographed on four sides after test only. The middle of the length of the beam, the load and support points, and the order of occurrence of the failures shall be indicated so as to show in the photograph; if necessary to bring them out in the photograph the failures should be penciled or pointed.

MOISTURE CONTROL

Control of Moisture Content

18. (*a*) As specified in Section 14 (*c*), the major specimens to be tested in the air-dry condition shall be brought to practically constant weight before test. All tests shall be carried out in such a manner as to prevent large changes in moisture content.

(*b*) All minor specimens shall be tested immediately after cutting from the majors to prevent change in moisture content.

STATIC BENDING TEST OF STRUCTURAL SIZE TIMBER

Size and Weight

19. (*a*) The beam shall be weighed before test. The length and the actual depth and width at the center shall be measured.

[6] The procedure outlined utilizes to best advantage the entire major specimen and if carefully followed will not usually admit the inclusion of specimens which have been seriously strained in the major bending test. As specified in Section 26 of the Standard Methods of Testing Small Clear Specimens of Timber (A.S.T.M. Designation: D 143), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book, the minors selected shall be free from defects which will affect the properties under consideration.

(*b*) For nonrectangular beams or beams nonuniform in section, data shall be secured from which the exact size and shape of the section at any point throughout the length may be obtained.

Additional Marking

20. In addition to the markings previously made, the four faces shall be lettered "*a*," "*b*," "*c*," and "*d*," beginning with *a* for the face to which the load is applied and proceeding successively to the right (clockwise) when viewed from the "*A*" or butt end of the timber.

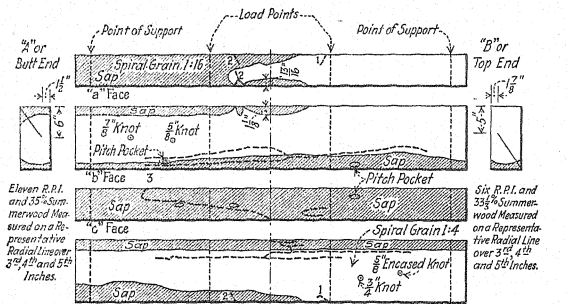


FIG. 1.—Sketch of Structural Size Timber Showing Method of Recording Location and Type of Defects and Other Pertinent Data.

Broken lines indicate test failures, and the accompanying numbers, their order of occurrence.

Sketch of Beam

21. As part of the record, a sketch shall be made of the faces and ends of each beam showing the size, location, and type of defects, including cross-grain,[7] the distribution of sapwood and heartwood, the location of pith, when present, and the direction of the annual rings (see Fig. 1).[8] Detailed record shall also be made of other defects which may be present, such as crook, bow, cup, and twist.[9] The number of rings per inch and proportion of summerwood at both ends shall also be recorded.

Loading, Span, and Supports

22. (*a*) For timbers in structural sizes such as bridge stringers and joists, third-point loading over a span length of 15 ft. shall be used. Knife-edges rigidly attached to an auxiliary beam which in turn is hinged by a knife-edge shall be used in applying the load. The supporting knife-edges shall preferably be of the half-rocker type, and shall be so placed as to rock outward. To prevent accident or damage from their being thrust suddenly outward on failure of the specimen, they should be tied together by means of a slack chain or cable. Metal bearing plates 6 in. in

[7] Cross-grained wood is that in which the wood cells or fibers do not run parallel with the axis of the piece. Cross-grain may be classified as spiral, diagonal, dip, wavy, curly, and interlocking. Definitions of these terms may be found in the Standard Definitions of Terms Relating to Timber (A.S.T.M. Designation: D 9), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book. The presence of diagonal and spiral grain is usually indicated by surface checks; these invariably follow the direction of the grain. The amount of cross-grain in any portion of a timber may be expressed by the slope of the fibers (or checks) on each face with respect to the edges of the piece. Thus, a slope of 1 in 10 indicates a departure of the fibers from the direction of the axis of the specimen of 1 in. in 10 in. of length.

[8] Cross-section paper or quadrille-ruled note book paper is recommended for conveniently making this sketch of the beam to scale.

[9] For definitions of these terms, see U. S. Department of Agriculture *Circular No. 295*, p. 65.

width and not less than $\frac{1}{2}$ in. in thickness and extending entirely across the face of the beam shall be used over the supporting knife-edges. (See Fig. 2.)

(*b*) When beams are tested with respect to particular uses, the dimensions shall be such as to simulate actual conditions as closely as practicable.

(*c*) Fabricated beams such as I or box types shall be full size or solid at the loading points without abrupt change from the solid to the normal beam section.

(*d*) Center loading shall not be used for beams over 4 in. in depth when the span-depth ratio is 14 or less.

specified for third-point loading under Paragraph (*a*).

(*c*) Whenever possible, span-depth ratios between 11 and 15 shall be used. For span-depth ratios less than 14, the radius of curvature of the bearing blocks shall be proportionally increased over that specified in Paragraphs (*a*) and (*b*).

(*d*) Bearing blocks of the form and size shown in Fig. 3 shall be used at the load points in testing timbers in structural sizes, such as 6 by 12-in. and 8 by 16-in. bridge stringers, on a 15-ft. span.

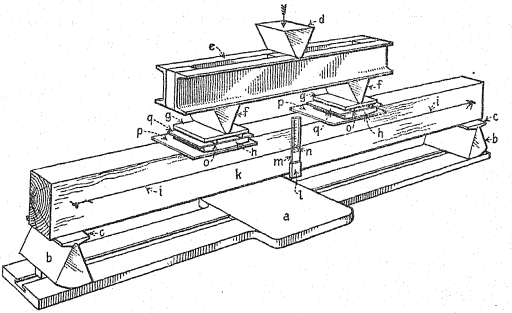


FIG. 2.—Diagrammatic Sketch Showing Method of Conducting Static Bending Test of Structural Sizes of Timber.

Bearing Blocks

23. (*a*) When testing beams under third-point loading on a span equal to 14 times the beam depth the load shall be applied through bearing blocks extending entirely across the face of the beam and having a radius of curvature three times the depth of the beam for a chord length at least equal to the depth of beam. Additional bearing may be obtained by continuing the curvature in both directions at a radius equal to twice the depth of the beam.

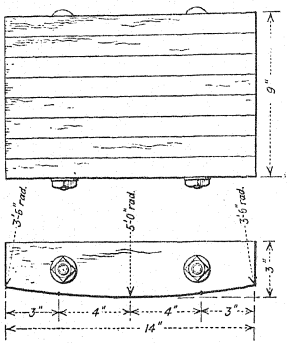
(*b*) For beams loaded at the center, the radius of the bearing block shall be one-half that

(*e*) When the depth of beam is greater than 4 in., a thin steel plate slightly wider than the beam shall be placed between the loading block and the beam. For beams up to 6 in. in depth, the thickness of this plate shall be from $\frac{3}{32}$ to $\frac{1}{8}$ in. For greater depth the thickness of plates shall be increased slightly to about $\frac{5}{32}$ in. for timbers 6 by 12 in. or 8 by 16 in. in cross-section.

(*f*) For fabricated sections such as airplane I and box beams up to 6 in. in depth, the plate shall be from $\frac{1}{16}$ to $\frac{3}{32}$ in. in thickness.

(*g*) Metal bearing plates and rollers shall be used between each bearing block

and its corresponding knife-edge. The plates between rollers and knife-edges shall be not less than $1\frac{1}{4}$ in. in thickness. If the supporting knife-edges are of the full-rocker type rather than the half-rocker type recommended in Section 22 (*a*), rollers shall be placed under one loading knife-edge only.



Hard Maple
Laminated Construction—Glued and Bolted.

FIG. 3.—Details of Bearing Block for Timbers 12 to 16 in. in Depth When Tested in Third-Point Loading over 15-ft. Span.

Placement of Specimen in Machine

24. The beam shall be placed in the testing machine symmetrically with respect to the supports. Parallelism shall be obtained at bearing points. In the case of warped beams, wedges or shims shall be used between the plate and the loading or supporting knife-edges or both, as may be necessary to obtain an even bearing before the load is applied.[10] In the case of timbers containing defects, the load shall be applied to the poorer of the loading faces.

[10] The use of shims or wedges is recommended in preference to planing because in certain cases the latter procedure may effect changes in section where undesirable.

Speed of Testing

25. (*a*) The load shall be applied continuously, and with a uniform motion of the movable head throughout the test.

(*b*) *Case 1.*—Beams 4 in. or less in depth when containing defects, or 8 in. or less in depth when free from defects, shall be tested at a rate of fiber strain of 0.0015 in. per inch of outer fiber length per minute.[11]

Case 2.—Beams over 4 in. in depth when containing defects, or over 8 in. in depth when free from defects, shall be tested at a rate of fiber strain of 0.0007 in. per inch of outer fiber length per minute.[11]

(*c*) For two symmetrical concentrated loads the rate of motion of the movable head of the testing machine may be calculated from the formula:

$$N = \frac{z}{3}\frac{a}{d}(3L - 4a)$$

where:

N = rate of motion of moving head in inches per minute,
z = unit rate of fiber strain in inches per inch of outer fiber length per minute,
a = distance from support to adjacent load in inches,
d = depth of beam in inches, and
L = span in inches.

For example, in testing beams 16 in. in depth under third-point loading and on a 15-ft. span, the rate of motion of movable head shall be 0.26 ± 0.065 in. per min.[9]

(*d*) For center loading, the rate of motion of the movable head of the testing machine may be calculated from the formula:

$$N = \frac{z L^2}{6 d}$$

where the same legend applies as under Paragraph (*c*).

[11] For permissible variation in rate of motion of movable head see Section 51.

Load-Deflection Curves

26. (*a*) Load-deflection curves shall be plotted throughout the test.[12] The load and deflection at first failure, at the maximum load, and at points of sudden change shall be shown on the curve, although they may have to be interpolated because of not occurring at one of the regular load or deflection increments.

(*b*) The test shall be continued until the maximum load has been well passed. In the event of horizontal shear, the maximum load may occur after such failure.

Method of Determining Deflection

27. (*a*) Deflections of the beam at mid-height at the center of the span with respect to points at mid-height immediately above the supports shall be taken to the nearest 0.01 in.

(*b*) Deflection may be read directly by means of a wire stretched between nails driven at mid-height of the beam immediately above the supports and extending across a scale attached at mid-height at the center of the span. Figure 2 illustrates the method of test. A telescope or reading glass will be found useful in observing the deflections. When a reading glass is used parallax shall be prevented by placing a reflecting surface adjacent to the scale in order that the wire and its image may be made to coincide.

(*c*) Where special refinement is desired, deflection readings may be taken simultaneously on both sides of the beam, the wires (Paragraph (*b*)) being supported over free running pulleys and held taut with small suspended weights.

Description of Failures

28. The failures shall be described in detail as to type, manner and order of occurrence, and position in beam. The descriptions of the failures shall be suitably recorded and correlated with the load-deflection curves. The failures shall be sketched on the drawing of the beam referred to in Section 21, with notations as to the order of their occurrence. The section of the beam containing the failure shall be held for examination and reference until analysis of the data has been completed.

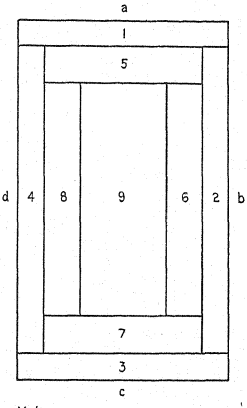


FIG. 4.—Cross-Section of Timber Showing Method of Cutting Up Moisture Distribution Sections.

Identification Sample

29. A section of the beam at least 2 in. in length shall be retained for purposes of identification and future reference. The number of rings per inch and proportion of summerwood shall be recorded for this section.

[12] See Fig. 1 of the Appendix for a sample static-bending data sheet and Fig. 2 of the Appendix for a sample computation data card.

Specimens for Moisture Determinations

30. Two sections 1 in. in length shall be cut from near the place of failure for moisture determinations. One of these shall be used for determining the average moisture content of the test specimen. The other, which may be termed a moisture-distribution section, shall be used for determining the moisture content of different portions of the cross-section and shall be sawed into parts according to the pattern shown in Fig. 4. Moisture determinations in each case shall be made in accordance with the procedure specified in Sections 44 to 48.

Loading

32. The load shall be applied through a metal plate 6 in. in width placed across a clear face of the specimen at the center of the length and at right angles to it. The load shall be applied to a face which corresponds to either the loading or supporting face of the static bending specimen. (Section 24.) (See Fig. 5.)

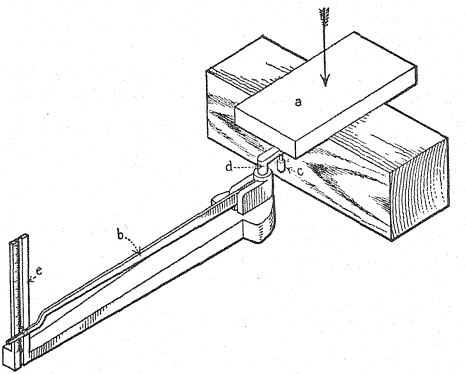


FIG. 5.—Diagrammatic Sketch of Method of Conducting Compression-Perpendicular-to-Grain Test.

COMPRESSION PERPENDICULAR TO GRAIN

Size of Specimen

31. The compression-perpendicular-to-grain tests shall be made on specimens 30 in. in length and of the cross-section of the major beam. The actual width and length shall be measured and recorded, and all defects shall be described.

Speed of Testing

33. (*a*) The load shall be applied continuously throughout the test at a rate of motion of movable head[11] calculated from the formula:[12]

$$N = 0.0175\, d^4$$

where:

N = rate of motion of moving head in inches per minute, and

d = depth of beam in inches.

[12] Tests are now in progress to establish more definitely the proper rate of application of load; therefore, the requirement of this section may be changed on the completion of the study.

(*b*) For the specimens 8 by 16 in. in cross-section, for example, the rate of descent of the movable head shall be 0.060 ± 0.015 in. per min.[11]

Load-Compression Curves

34. Load-compression curves[14] shall be taken for all specimens until the elastic limit has been well passed, after which the test shall be discontinued. Compression readings shall be taken to 0.001 in.

Weight and Moisture Content

35. The specimen shall be weighed after test, and two sections shall be cut near the place of failure for moisture determinations. One of these shall be used for determining the average moisture content of the specimen. The other, which may be termed a moisture distribution section, shall be used for determining the moisture content of different portions of the cross-section and shall be sawed into parts according to the pattern shown in Fig. 4. Moisture determinations in each case shall be made in accordance with the procedure specified in Sections 44 to 48.

COMPRESSION PARALLEL TO GRAIN

Size of Specimen

36. The major compression-parallel-to-grain tests shall be made on the 6 by 6 by 24-in. specimens prepared from the pieces specified in Section 15 (*c*). The actual cross-section dimensions, the rate of growth, and the percentage of summerwood shall be determined and recorded for each specimen. All defects shall be described.

End Surfaces

37. Care shall be exercised in preparing the compression-parallel-to-grain test specimens to make the end surfaces plane, parallel, and normal to the axis of the specimen.[15]

Spherical Bearing

38. (*a*) A spherical bearing block shall be used to prevent eccentricity of loading. The radius of the sphere shall be as small as practicable, in order to facilitate adjustment of the bearing plate to the specimen. The suspended type of spherical bearing, to be used on top of the specimen, is especially recommended. The size of the compression plate shall be slightly larger than is necessary to encompass the end of the specimen when the latter is centered on the head.

(*b*) The adjustment of the suspended spherical bearing block may be accomplished by keeping the compression plate in motion about its bearing as contact is made with the specimen.

(*c*) In the spherical bearing block for use on the weighing table, adjustment shall be made by moving the bearing plate with the specimen centered thereon.

Speed of Testing

39. The load shall be applied continuously throughout the test at a rate of motion of the movable head of 0.036 ± 0.009 in. per min.[11]

Records

40. The maximum load obtained shall be recorded, together with a description and sketch of the failure. The section of the specimen containing the failure shall be held for examination and refer-

[14] See Fig. 4 of the Appendix for a sample compression-perpendicular-to-grain data sheet and Fig. 3 of the Appendix for a sample computation data card.

[15] A sharp fine-toothed saw of either the cross-cut or "novelty" cross-cut type is indispensable for obtaining smooth end surfaces. Power equipment with accurate table guides is especially recommended for this work, though miter-box hand sawing is permissible. Hand planing of the ends, if carefully done, is sometimes advisable.

ence until analysis of the data has been completed.

Position of Test Failures

41. In order to obtain satisfactory and uniform results, the failures shall be made to develop in the body of the specimen. With specimens of uniform cross-section this may be obtained by having the ends properly prepared and dried to a slightly lower moisture content than the body. With green material, it will usually suffice to close-pile the specimens, cover the body with a damp or wet cloth, and expose the ends for a short time. For air-dry material it may sometimes be advisable, should the failures in test indicate that a slight end drying is necessary, to pile the specimens in a similar manner and place them in a desiccator.

Weight and Moisture Content

42. The specimen shall be weighed after test, and a moisture section shall be cut from the body of the piece near the failure. Moisture determinations shall be made in accordance with the procedure specified in Sections 44 to 48.

MINOR TESTS

Minor Test Procedures

43. The procedure for the conducting of minor tests shall conform to that prescribed in the Standard Methods of Testing Small Clear Specimens of Timber (A.S.T.M. Designation: D 143) of the American Society for Testing Materials.[16]

MOISTURE DETERMINATIONS

Selection of Samples

44. The samples for moisture determinations shall be selected as hereinbefore described for each test.

[16] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Cutting of "Distribution" Sample

45. The moisture-distribution specimens shall be cut into sections according to Fig. 4, as specified in Sections 30 and 35.

Weighing of Moisture Sample

46. Immediately upon obtaining any moisture sample, all loose splinters shall be removed and the sample weighed.

Drying

47. The moisture samples shall be open-piled in a ventilated oven and dried at a temperature which shall ultimately be increased to 98 ± 3 C. until constant weight is attained, after which the resulting weight shall be carefully determined and recorded as the oven-dry weight. For specimens not over 1 in. in length (in the direction of grain) continuous drying for about 48 hr. under the conditions specified is sufficient to bring the specimens to constant weight. For drying longer specimens, considerably more time will be required, since the rate of drying from the side grain surface of wood is only about one-sixth that from the end surface.

Moisture Content

48. The loss in weight expressed as a percentage of the oven-dry weight as above determined shall be considered the moisture content of the specimen.

PERMISSIBLE VARIATIONS

Weights

49. The weight of test specimens and of moisture samples shall be determined within 0.2 per cent.

Measurements

50. Measurements of test specimens shall be made to the nearest 0.01 in.

Testing Machine Speeds

51. In no case shall the testing machine speed used vary more than 25 per cent[17] from that specified for a given test. The load shall be applied continuously at the required speed throughout the test.

[17] Tests by the U. S. Forest Service show that this speed limitation is necessary in order that the stress shall not vary more than 1 per cent due to variation in rate of fiber strain.

CALIBRATION

Calibration

52. All apparatus used in obtaining data shall be calibrated at sufficiently frequent intervals to insure accuracy.[18]

[18] See the Standard Methods of Verification of Testing Machines (A.S.T.M. Designation E 4), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(See Appendix, p. 774)

APPENDIX

Timber Test Log Sheet

U. S. DEPARTMENT OF AGRICULTURE
FOREST SERVICE

Project No. 164
Working Plan No. 184
Laboratory No. 123847
Station Madison
Date Nov. 5, 1915
Ship. No. I-308 Stick No. ——
Piece No. 30 Mark ——

Species Longleaf Pine
Kind of test Static Bending
Grade Extra Select (S1)
Group ——
Loading Third Point
Span 180 in.
Distance between collars ——
Width of plate ——
Machine M-1041
Speed of motor, 0.26 in. per min.
Weight of hammer ——
Height 15.61 in.
Width 7.65 in.
Length 16.00 Ft.
Cross-section ——
Weight 515 lb.
Rings per inch 7-7-10-13-17-24-32-29-20
Slip 0
Summerwood 24-34-33-37-30-37-34-32-40
Seasoning Air Dry
Moisture (7.8 % (Average)
Kind of failure Horizontal Shear Followed by Compression and Tension.
Remarks Timber of Medium Density and relatively free from Defects except Small Checks along One Side.
Sketch See Separate Sheet

Note: Values for Rings per Inch and Percent Summerwood are those for each Successive Inch from Edge to Edge of the Timber along an Avarage Radial Line.

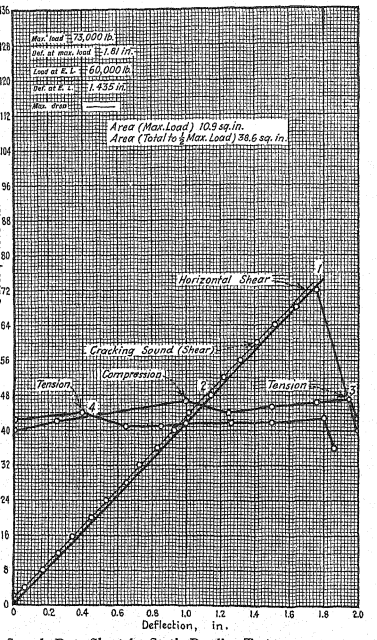


FIG. 1.—Sample Data Sheet for Static Bending Test.

Form 507.
(Revised January, 1918.)

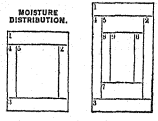
STATIC BENDING

5-308 (Ship No.) (Stick No.) One third points. Loading 123647 (Lab. No.)
30 (Piece No.) (Mark.) Station MADISON, WIS. Date 11-5-15 184 (Project No.)
Species Longleaf Pine Grade No 1 Seasoning Air dry
Rings 7-7-10-13-17-24-32-22-20 Sap 0 % Summerwood 34-24-33-37-40-37-34-32-40 % Moisture 17.2 %
Span 180 in. Length 15.00 ft. Height 15.01 in. Width 2.85 in. Weight 5.15 ¼ #

SPECIFIC GRAVITY. AS TEST.	SPECIFIC GRAVITY. OV. DRY.	F. S. AT E. L.	M. OF R.	M. OF E.	SHEAR.	WORK TO E. L.	WORK TO MAX. LOAD.	TOTAL WORK.	
.622	.528	5820	7080	1781	458	174	3.2	11.5	

Rings: Up. ¼ ——— Mid. ¼ ——— Low. ¼ ———
Sum. wood: Up. ¼ ——— Mid. ¼ ——— Low. ¼ ———
Defects ———
Failure Horizontal Shear followed by Compression and tension.

MOISTURE DISTRIBUTION.



8—1104
19798 M

FIG. 2.—Sample Computation Data Card for Static Bending Test.

(Revised January, 1918)

COMPRESSION AT RIGHT ANGLES TO GRAIN

308 (Ship No.) ——— (Stick No.) 124509 (Lab. No.)
30 (Piece No.) ——— (Mark.) Station MADISON, WIS. Date 11-30-15 184 (Project No.)
Species Longleaf Pine Grade 1 Seasoning Air dry
Rings 20 Sap 0 % Summerwood 35 % Moisture 17.4 %
Width of plate 2.98 Length 30.11 in. Height 15.56 in. Width 7.51 in. Weight 76 #

SPECIFIC GRAVITY		LOAD AT E. L.	CRUSH. ST. AT E. L.	Δ ÷ h	
At Test.	Ov. Dry				
.598	.510	38,000 #	854	———	

8—1404
19798 M

FIG. 3.—Sample Computation Data Card for Compression-Perpendicular-to-Grain Test.

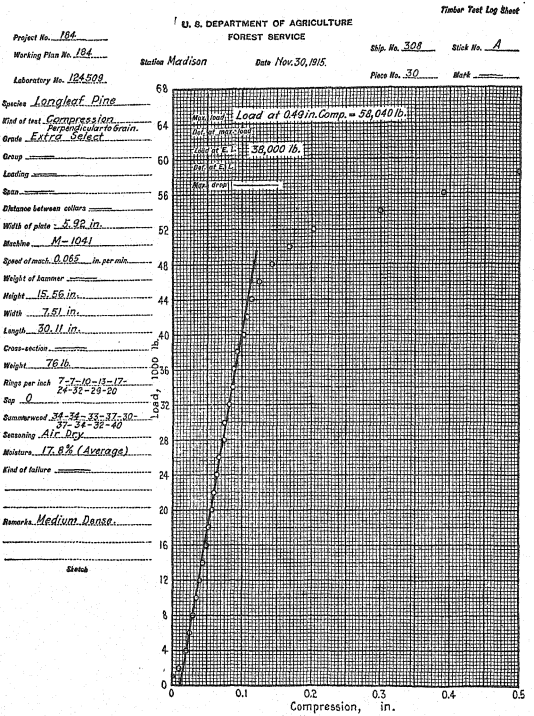


FIG. 4.—Sample Data Sheet for Compression-Perpendicular-to-Grain Tests.

Standard Definitions of

TERMS RELATING TO TIMBER[1]

A.S.T.M. Designation: D 9 - 30

ADOPTED, 1907; REVISED, 1915, 1930.

This Standard of the American Society for Testing Materials is issued under the fixed designation D 9; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Structural Timber.—By the term "Structural Timber" is understood such products of wood in which the strength of the timber is the controlling element in its selection and use, such as trestle timbers (stringers, caps, posts, mud sills, bracing, bridge ties, guard rails); car timbers (car framing, including upper framing, car sills); framing for buildings (posts, mud sills, girders, framing joists); ship timbers (ship timbers, ship decking); and cross arms for poles.

TERMS RELATING TO WOOD

Annual Ring.—In the case of wood, the growth layer put on in a single growth year.

Heartwood.—The inner, dead portion of the wood in a tree.

NOTE.—It is usually dark in color. In trees like white spruce, hemlock, white fir, basswood, and holly, the heartwood is not distinctly darker than the sapwood, and the two can not be readily distinguished.

Sapwood.—The outer, live portion of the wood in a tree.

NOTE.—It is usually light in color.

Springwood.—The softer, more porous portion of each annual ring.

Summerwood.—The harder, less porous portion of each annual ring.

NOTE.—In softwoods it is darker than the springwood, but not always so in hardwoods. If the annual rings are fairly uniform in texture, as in maple, red gum, and yellow poplar, no distinction can be made between springwood and summerwood.

Pith.—In the case of wood, the small soft core occurring in the structural center.

Sound Wood.—Wood free from any form of decay, incipient or advanced.

STANDARD DEFECTS

Methods of measurement of size of knots or holes in the use-classifications of joist and plank, beams and stringers, and posts and timbers, are given in the Standard Specifications for Structural Wood Joist and Plank, Beams and Stringers, and Posts and Timbers (A.S.T.M. Designation: D 245) of the American Society for Testing Materials.[2]

Defect.—In the case of wood, any irregularity occurring in or on the wood that may lower its strength.

Blemish.—In the case of wood, anything, not necessarily a defect, marring its appearance.

[1] Under the standardization procedure of the Society, these definitions are under the jurisdiction of the A.S.T.M. Committee D-7 on Timber.

[2] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Bark Pocket.—Bark partially or wholly enclosed in wood.

Check.—In the case of wood, a separation along the grain, the greater part of which occurs across the rings of annual growth.

End Check.—A check occurring at an end of a piece.

Heart Check.—A check starting near the pith and extending toward but not to the surface of a piece.

NOTE.—Several of these occurring together are called a star check.

Surface Check.—A check occurring at the surface of a piece.

Through Check.—A check extending through the piece from one surface to an opposite or to an adjoining surface.

Collapse.—In the case of wood, a caving at a surface of a piece.

NOTE.—It sometimes occurs in streaks, giving the surface a corrugated appearance and often is due to the flattening of the cells when wet wood is dried quickly.

Honeycombing.—In the case of wood, checks that occur in the interior of a piece, often not visible at the surface.

NOTE.—On a cross-section they usually appear as slits or pockets the width of which may be very large relative to the radial length.

Cross Break.—A separation of the wood cells across the grain.

NOTE.—Such breaks may be due to internal strains resulting from unequal shrinkage or to external forces.

Cross-Grained Wood.—Wood in which the fibers are not parallel with the axis of a piece.

Curly-Grained Wood.—Wood in which the fibers are distorted so that they afford a curled appearance, as in "bird's eye" wood.

NOTE.—Areas showing curly grain may vary up to several inches in diameter.

Diagonal-Grained Wood.—Wood in which the fibers are at an angle with (that is, diagonal to) the axis of a piece as a result of sawing at an angle with the axis of the tree.

NOTE.—It may appear on either the radial or flat-grain surface.

Dip-Grained Wood.—Wood which has single waves or undulations of the fibers, such as occur around knots and pitch pockets.

Interlocked-Grained Wood.—Wood in which the fibers are inclined in one direction in a number of rings of annual growth, then gradually reverse and are inclined in an opposite direction in succeeding growth rings, then later again reverse, etc.

Spiral-Grained Wood.—Wood in which the fibers take a more or less winding or spiral course, as in a twisted tree.

NOTE.—It may be detected on the flat grain surface.

Wavy-Grained Wood.—Wood in which the fibers collectively take the form of waves or undulations.

NOTE.—It may appear on either the radial or flat-grain surface and is indicated by the wavy surface of a split piece.

Decay.—In the case of wood, destruction of the wood substance due to the action of wood-destroying fungi.

NOTE.—"Dote" and "rot" are synonymous with "decay" and are any form of decay which may be evident either as a dark red discoloration not found in the sound wood, or the presence of white or red rotten spots.

Advanced (or Typical) Decay.—In the case of wood, the older stage of decay in which the destruction is readily recognized because the wood has become punky, soft and spongy, stringy, ring-shaked, pitted, or crumbly.

NOTE.—Decided discoloration or bleaching of the rotted wood is often apparent as for example, brown and white rots, pocket rots.

Incipient Decay.—In the case of wood, the early stage of decay which has not proceeded far enough to soften or otherwise perceptibly impair the hardness of the wood.

NOTE.—It is usually accompanied by a slight discoloration or bleaching of the wood as instanced by the "firm red heart" in softwoods and the "water-soak" stage in certain types of decay.

Firm Red Heart.—A stage of incipient decay in wood characterized by a reddish color produced in the heartwood, which does not, however, make the wood unfit for the majority of yard purposes.

NOTE.—It is caused by the fungus *Trametes pini* and occurs chiefly in the pines.

Water-Soak (or Stain).—A term applied to a generally water-soaked area in heartwood, which is usually interpreted as the incipient stage of certain wood decays.

NOTE.—It occurs in hemlock and possibly in other woods.

Knot.[2]—In the case of wood, that portion of a branch which has become incorporated in the body of a tree.

Pin Knot.—A knot not over ½ in. in diameter.

Small Knot.—A knot over ½ in., but not over ¾ in., in diameter.

Medium Knot.—A knot over ¾ in., but not over 1½ in., in diameter.

Large Knot.—A knot more than 1½ in. in diameter.

Encased Knot.—A knot whose rings of annual growth are not intergrown and homogeneous with those of the surrounding wood.

NOTE.—The encasement may be partial or complete; if intergrown partially or so fixed by growth or position that it will retain its place in the piece, it shall be considered a tight knot; if completely intergrown on one face, it is a watertight knot.

Intergrown Knot.—A knot whose rings of annual growth are completely intergrown with those of the surrounding wood.

Loose Knot.—A knot not firmly held in place by growth or position.

Tight Knot.—A knot so fixed by growth or position that it will firmly retain its place in the piece.

Pith Knot.—A sound knot with a pith hole not more than ¼ in. in diameter in the center.

Round Knot.—A knot whose sawn section is oval or circular.

Spike Knot.—A knot sawn in a lengthwise direction.

Decayed Knot.—A knot which due to advanced decay is not as hard as the surrounding wood.

Sound Knot.—A knot which is solid across its face and which is as hard as the surrounding wood.

NOTE.—Red or black knots may be sound.

Pitch Pocket.—An opening between the grain of the wood, containing more or less pitch.

NOTE.—Pitch pockets are classified as small, medium and large.

Small Pitch Pocket.—A pocket not over ⅛ in. in width or not over 4 in. in length, or not over ¼ in. in width and not over 2 in. in length.

Medium Pitch Pocket.—A pocket not over ⅜ in. in width and 4 in. in length, or not over ⅛ in. in width and not over 8 in. in length.

Large Pitch Pocket.—A pocket over ⅜ in. in width and over 4 in. in length, or over ⅛ in. in width and over 8 in. in length.

Pitch Streak.—A well-defined accumulation of pitch at one point in the piece.

NOTE.—When not sufficient to develop a well-defined streak, or where the fiber between grains, that is, the coarse-grained fiber, usually termed "springwood," is not saturated with pitch, it is not considered a defect.

Shake.—In the case of wood, a separation along the grain, the greater part of which occurs between the rings of annual growth.

Through Shake.—A shake which extends between any two faces of a timber.

Wane.—Bark or lack of wood from any cause on edges of timbers.

[2] Information covering the interpretation of knot measurements is given in the appendix, see p. 781.

STANDARD NAMES FOR STRUCTURAL TIMBERS

Commercial Name	Botanical Name
Cedars:	
Alaska Cedar	Chamaecyparis nootkatensis
Northern White Cedar	Thuja occidentalis
Port Orford Cedar	Chamaecyparis lawsoniana
Southern White Cedar	Chamaecyparis thyoides
Western Red Cedar	Thuja plicata
Cypress:	
Red Cypress	Taxodium distichum (Coast type)
Yellow Cypress	Taxodium distichum (Inland type)
White Cypress	Taxodium distichum (Inland type)
Douglas Fir:	
Douglas Fir	Pseudotsuga taxifolia (Coast type)
Red Fir	Pseudotsuga taxifolia (Intermountain type)
Red Fir	Pseudotsuga taxifolia (Rocky Mountain type)
The True Firs:	
Balsam Fir	Abies balsamea Abies fraseri (Southern balsam fir)
Golden Fir	Abies magnifica
Noble Fir	Abies nobilis
Silver Fir	Abies amabilis
White Fir	Abies concolor Abies grandis (lowland white fir)
Hemlocks:	
Eastern Hemlock	Tsuga canadensis Tsuga caroliniana (Carolina hemlock)
West Coast Hemlock	Tsuga heterophylla (Western hemlock)
Larch:	
Western Larch	Larix occidentalis
Pines:	
California White Pine	Pinus ponderosa (Western yellow pine) Pinus jeffreyi (Jeffrey pine)
Idaho White Pine	Pinus monticola
Northern White Pine	Pinus strobus
Norway Pine	Pinus resinosa
Pondosa Pine	Pinus ponderosa (Western yellow pine)
Southern Pine (Southern Yellow Pine)	Pinus taeda (loblolly pine) Pinus palustris (longleaf pine) Pinus serotina (pond pine) Pinus echinata (shortleaf pine) Pinus caribaea (slash pine) Pinus rigida (pitch pine) Pinus glabra (spruce pine)
Sugar Pine	Pinus lambertiana
Redwood:	
Redwood	Sequoia sempervirens
Spruces:	
Eastern Spruce	Picea mariana (black spruce) Picea rubra (red spruce) Picea glauca (white spruce)
Engelmann Spruce	Picea engelmanni Picea parryana (blue spruce)
Sitka Spruce	Picea sitchensis
Tamarack:	
Tamarack	Larix laricina

APPENDIX[4]

THE SIZE OF A KNOT

(Supplementing Definition of a Knot)

The A.S.T.M. standards for timber give very definite knot sizes that are to be permitted in any given grade and size of timber and a very concise and accurate definition of a knot. But it is not always easy to trace the outline of the knot on the surface of a timber and segregate the knot from the cross grain around the knot, which is a part of the body of the tree.

Sometimes there is a difference in color between the end grain of the limb wood and the surrounding wood. Sometimes there is a marked change in color that merely marks the heartwood of a live knot, and the sapwood of the knot may have the same color as the surrounding wood.

Again, we find knots in which there is practically no difference in color. The body of the tree is usually enlarged at a knot so that, when a knot is sawed through, the ring growth of the body wood looks very much like a part of the knot. How then are knot sizes to be determined?

There are two ways of determining the limits of a live knot when color or general appearance does not clearly demonstrate the boundary of the knot:

First.—The rings on the top of a limb are usually narrower than those on the bottom; and on a tangentially cut surface these rings at the top are narrower than those showing immediately above the knot in the body of the tree; also the rings in the body of the tree get wider as you measure away from the knot. The growth ring at the top of the knot can be traced around the knot to outline the size of the knot. When the knot is cut at an angle there will, as a rule, still be a place to one side of the top where the relatively narrow ring growth of the knot is suddenly changed

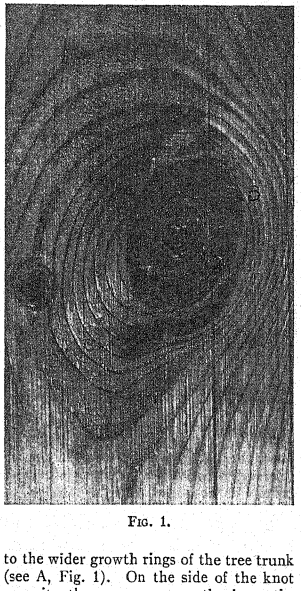


FIG. 1.

to the wider growth rings of the tree trunk (see A, Fig. 1). On the side of the knot opposite these narrow growth rings, the growth rings will usually be found to get wider all the way from the pith center out

[4] Prepared by John A. Newlin, Specialist in the Mechanics of Wood, Forest Products Laboratory, Forest Service, U. S. Department of Agriculture, maintained at Madison, Wis., in cooperation with the University of Wisconsin.

and it is often very difficult to establish the limit of the knots on that side except by tracing the growth rings from the other side of the knot.

Second.—Checks on the face of a knot run radially from the pith center and those running to the sides never run beyond the knot without an abrupt change in direction.

To one trying to determine the size of a knot, the oaks give a great deal of trouble due to two principal causes: First, color cannot be depended upon to outline the knot, and second, the body of the oak tree is usually greatly enlarged at a limb and a large amount of irregular or burly growth is introduced by a cut through the knot.

On the top of the oak knot it will usually be found that the very definite growth rings of the knot change very abruptly to burly irregular grain (see A, Fig. 2), often with bark pockets (see A, Fig. 3). This is the body wood above the knot. The radial checking in the oak knot is often very prominent and can be used to determine the limits of the knot. The checks follow the rays and run approximately radially from the pith center of the knots. The checks running to the bottom of the knot may run approximately straight far beyond the limit of the knot and occasionally those running to the top may run through the burly wood above the knot in more or less of a straight line, but the checks running to the sides of the knot, while they may stop short of the limit of the knot, never run without an abrupt change of direction beyond the limit of the knot (see B, Figs. 1 and 2).

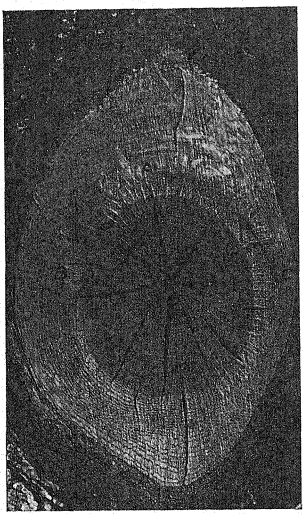


FIG. 2.



FIG. 3.

Standard Specifications for
CREOSOTE[1]

A.S.T.M. Designation: D 390 – 36

ADOPTED, 1935; REVISED, 1936.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 390; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover creosote for use in preservative treatment of timber, as defined in the Standard Definitions of Terms Relating to Timber Preservatives (A.S.T.M. Designation: D 324) of the American Society for Testing Materials.[3] The creosote shall be a distillate of coal-gas or coke-oven tar.

Properties

2. Creosote shall conform to the following requirements:

Water, max., per cent	3
Matter insoluble in benzol[a], max., per cent	0.5
Specific gravity, 38 C./15.5 C., min.	1.03
Distillation, based on water-free oil, max., per cent:	
Up to 210 C.	5
Up to 235 C.	25
Coke residue, max., per cent	2

[a] Samples of creosote taken from working tanks may show an increase in matter insoluble in benzol due to treating operations. Such increases, provided they do not exceed by 1 per cent the specification limits, should not serve to cause rejection of the creosote for nonconformity with the specifications, if it can be shown that the original fresh oil was of specified quality.

NOTE.—Owing to the complexity of the chemical composition and physical properties of coal tar creosote, and to the fact that some of the same compounds and properties which characterize coal tar creosote are found in certain petroleum derivatives, determination of the purity of creosote is difficult. When there is not certain assurance that the oil is a pure product, the following tests will aid in arriving at an opinion as to its coal tar origin:

(*1*) The fraction distilling between 210 and 235 C. is usually solid or contains some solids when cooled to 25 C.

(*2*) All of the fractions up to 315 C. contain tar acids in varying amounts, usually at least 1 per cent, calculated on the amount of the fraction tested.

(*3*) The specific gravity of the fraction between 235 and 315 C. is usually not lower than 1.025 and the specific gravity of the fraction between 315 and 355 C. is usually not lower than 1.085 at 38 C. compared with water at 15.5 C. However, some pure coal tar distillates fall slightly below these limits.

If the oil does not comply with at least one of the foregoing tests, it is undoubtedly not a pure coal tar creosote.

Sampling and Methods of Testing

3. The sampling and properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials:

(*a*) *Sampling.*—Standard Methods of

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-7 on Timber.

[2] Prior to adoption as standard, these specifications were published as tentative from 1934 to 1935.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Sampling and Testing Creosote (A.S.T.M. Designation: D 38).[3]

(*b*) *Water.*—Standard Method of Test for Water in Creosote (A.S.T.M. Designation: D 370)[3] or, as an alternate method, the Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (A.S.T.M. Designation: D 95).[3]

(*c*) *Matter Insoluble in Benzol.*—Standard Methods of Test for Insoluble Matter in Creosote (A.S.T.M. Designation: D 367).[3]

(*d*) *Specific Gravity.*—Standard Method of Test for Specific Gravity of Creosote (A.S.T.M. Designation: D 368).[3]

(*e*) *Distillation.*—Standard Method of Test for Distillation of Creosote (A.S.T.M. Designation: D 246).[3]

(*f*) *Specific Gravity of Fractions.*—Standard Method of Test for Specific Gravity, 38/15.5 C., of Creosote Fractions (A.S.T.M. Designation: D 369).[3]

(*g*) *Coke Residue.*—Standard Method of Test for Coke Residue of Creosote (A.S.T.M. Designation: D 168).[3]

(*h*) *Tar Acids.*—Standard Method of Test for Tar Acids in Creosote and Creosote – Coal Tar Solutions (A.S.T.M. Designation: D 453).[3]

(*i*) *Volume and Specific Gravity Correction.*—Standard Volume and Specific Gravity Correction Tables for Creosote and Coal Tar (A.S.T.M. Designation: D 347).[3]

Standard Specifications for
CREOSOTE-COAL TAR SOLUTION[1]

A.S.T.M. Designation: D 391 - 36

ADOPTED, 1935; REVISED, 1936.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 391; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover creosote-coal tar solution for use in preservative treatment of timber. The creosote-coal tar solution shall be a product of which at least 80 per cent shall be a distillate of coal-gas or coke-oven tar.

Properties

2. Creosote-coal tar solution shall conform to the following requirements:

Water, max., per cent	3
Matter insoluble in benzol,[a] max., per cent	2
Specific gravity, 38/15.5 C	1.05 to 1.12
Distillation, based on water-free oil, max., per cent:	
Up to 210 C	5
Up to 235 C	25
Coke residue, max., per cent	6

[a] Samples of solution taken from working tanks may show an increase in matter insoluble in benzol due to treating operations. Such increases, provided they do not exceed by 1 per cent the specification limits, should not serve to cause rejection of the solution for nonconformity with the specifications, if it can be shown that the original fresh oil was of specified quality.

NOTE.—Owing to the complexity of the chemical composition and physical properties of coal tar creosote, and to the fact that some of the same compounds and properties which characterize coal tar creosote are found in certain petroleum derivatives, determination of the purity of creosote is difficult. When there is not certain assurance that the oil is a pure product, the following tests will aid in arriving at an opinion as to its coal tar origin:

(*1*) The fraction distilling between 210 and 235 C. is usually solid or contains some solids when cooled to 25 C.

(*2*) All of the fractions up to 315 C. contain tar acids in varying amounts, usually at least 1 per cent, calculated on the amount of the fraction tested.

(*3*) The specific gravity of the fraction between 235 and 315 C. is usually not lower than 1.025 and the specific gravity of the fraction between 315 and 355 C. is usually not lower than 1.085 at 38 C. compared with water at 15.5 C. However, some pure coal tar distillates fall slightly below these limits.

If the oil does not comply with at least one of the foregoing tests, it is undoubtedly not a pure coal tar creosote.

Sampling and Methods of Testing

3. The sampling and properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials:

(*a*) *Sampling.*—Standard Methods of Sampling and Testing Creosote (A.S.T.M. Designation: D 38).[3]

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-7 on Timber.

[2] Prior to adoption as standard, these specifications were published as tentative from 1934 to 1935.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(*b*) *Water.*—Standard Method of Test for Water in Creosote (A.S.T.M. Designation: D 370)[2] or, as an alternate method, the Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (A.S.T.M. Designation: D 95).[3]

(*c*) *Matter Insoluble in Benzol.*—Standard Methods of Test for Insoluble Matter in Creosote (A.S.T.M. Designation: D 367).[3]

(*d*) *Specific Gravity.*—Standard Method of Test for Specific Gravity of Creosote (A.S.T.M. Designation: D 368).[3]

(*e*) *Distillation.*—Standard Method of Test for Distillation of Creosote (A.S.T.M. Designation: D 246).[3]

(*f*) *Specific Gravity of Fractions.*—Standard Method of Test for Specific Gravity, 38/15.5 C., of Creosote Fractions (A.S.T.M. Designation: D 369).[3]

(*g*) *Coke Residue.*—Standard Method of Test for Coke Residue of Creosote (A.S.T.M. Designation: D 168).[3]

(*h*) *Tar Acids.*—Standard Method of Test for Tar Acids in Creosote and Creosote – Coal Tar Solutions (A.S.T.M. Designation: D 453).[3]

(*i*) *Volume and Specific Gravity Correction.*—Standard Volume and Specific Gravity Correction Tables for Creosote and Coal Tar (A.S.T.M. Designation: D 347).[3]

Standard Specifications for

ZINC CHLORIDE[1]

A.S.T.M. Designation: D 432 - 39

ADOPTED, 1939.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 432; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover zinc chloride, either from commercial concentrated solutions of zinc chloride, or fused or granulated zinc chloride for use in the preservative treatment of timber.

Properties

2. (*a*) *Iron Content.*—The zinc chloride shall be acid-free and shall not contain more than 0.1 per cent of iron.

(*b*) *Chloride of Zinc Content.*—The material shall conform to the following minimum requirements as to percentage content of chloride of zinc:

	Chloride of Zinc, min., per cent
Concentrated zinc chloride solution	50
Fused or solid zinc chloride	94

Chemical Analysis

3. The material shall be analyzed in accordance with the Standard Methods of Chemical Analysis of Zinc Chloride (A.S.T.M. Designation: D 199) of the American Society for Testing Materials.[3]

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-7 on Timber.

These specifications are identical in substance with the American Wood-Preservers' Association Standard Specification for Zinc Chloride (No. 17a).

[2] Prior to adoption as standard, these specifications were published as tentative from 1936 to 1939.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Methods of

SAMPLING AND TESTING CREOSOTE[1]

A.S.T.M. Designation: D 38 – 33

ADOPTED, 1917; REVISED, 1918, 1924, 1927, 1930, 1933.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 38; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. (*a*) Creosote and creosote - coal tar solutions are homogeneous liquids, except for their moisture content. At atmospheric temperatures, however, part of the creosote is usually in crystalline form, and it is accordingly necessary before sampling that the creosote shall be heated to a temperature at which it is entirely liquid.

(*b*) When sampling creosote, the object is to obtain a sample that will be representative of the water content and the methods of sampling described or referred to in Sections 2 to 7 are intended to accomplish that purpose.

METHODS OF SAMPLING

Continuous Drip Sampling Method

Application

2. The continuous drip method of sampling is applicable wherever the creosote is being loaded or discharged from tank ships or barges by means of pumping.

Procedure

3. A ½-in. sampling pipe shall be inserted in the line through which the oil is being pumped, on the discharge side of the pump and preferably in a rising section of the pipe line. This sampling pipe shall extend one-half way to the center of the main pipe and with the inner open end of the sampling pipe turned at an angle of 90 deg. and facing the flow of the liquid. The sampling pipe shall be provided with a plug cock and shall discharge into a receiver of 50 to 100-gal. capacity. The plug cock shall be so adjusted that with a steady continuous flow of the oil the receiver shall be filled in the time required to pump the entire shipment. The receiver shall be provided with a steam coil sufficient to keep the contents at a temperature not exceeding 120 F. Immediately upon completion of the pumping, the contents of the receiver shall be very thoroughly mixed by agitation, rolling, or shaking, and duplicate 1-qt. samples taken immediately by means of a thief

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-7 on Timber.

[2] Prior to adoption as standard, these methods were published as tentative from 1915 to 1917, being revised in 1916 and 1917.

for purpose of tests. The amount of the drip sample collected shall be not less than 1 gal. for each 1000 gal. of oil, except in the case of large boat shipments, where a maximum of 100 gal. is sufficient.

Routine Zone Method of Sampling Tank Cars

Application

4. The zone sampling method is applicable to the routine sampling of creosote in tank cars. With suitable modification of the apparatus, this alternative or simplified method may also be applied to the sampling of creosote in storage tanks.

Apparatus

5. The sampling apparatus shall be a wide-mouthed vessel, attached to a wooden rod and fitted with a lid that can be withdrawn by means of a cord. The apparatus and markings on the rod shall be made substantially in accordance with the form and dimensions shown in Fig. 1.

Procedure

6. (*a*) The sampling apparatus shall first be lowered until the point marked "Zone No. 1" on the rod is level with the top of the dome of the tank car. The cover of the sampling can shall then be removed, the vessel allowed to fill at that point, and then slowly withdrawn. Extreme care shall be exercised in lifting the rod so as not to disturb the contents of the sampling vessel.

(*b*) Two samples shall be taken in a similar manner with the sampling apparatus lowered to the point marked "Zone No. 2," and one sample shall be likewise taken with the sampling apparatus lowered to the point marked "Zone No. 3."

(*c*) The four samples shall then be combined and mixed at once into a composite sample while the oil is thoroughly liquid. No sample shall be taken for analysis from oil which contains crystals.

Referee Method of Sampling Tank Cars

Procedure

7. In the case of dispute with reference to the water content of the shipment of creosote oil, sampling shall be carried out in accordance with the Standard Method of Sampling Creosote in Tank Cars of the American Wood Preservers' Association (Method No. 25*b*)[3] or of the American Railway Engineering Association.[4]

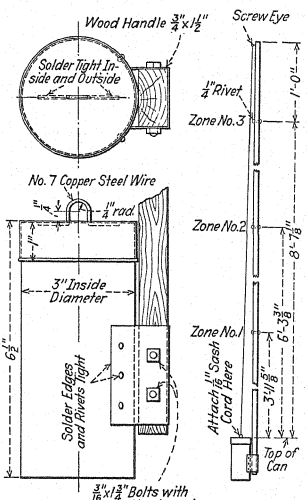


FIG. 1.—Routine Zone Sampling Apparatus.

[3] *Manual of Recommended Practice*, Am. Wood-Preservers' Assn.

[4] *Manual*, Am. Railway Engineering Assn., Chapter 17, p. 17 (1942).

METHODS OF TESTING

Methods of Testing

8. Creosote shall be analyzed and tested in accordance with the following methods of test of the American Society for Testing Materials:

(*a*) *Water.*—Standard Method of Test for Water in Creosote (A.S.T.M. Designation: D 370).[5]

(*b*) *Matter Insoluble in Benzol.*—Standard Methods of Test for Insoluble Matter in Creosote (A.S.T.M. Designation: D 367).[5]

(*c*) *Specific Gravity.*—Standard Method of Test for Specific Gravity of Creosote (A.S.T.M. Designation: D 368).[5]

(*d*) *Distillation.*—Standard Method of Test for Distillation of Creosote (A.S.T.M. Designation: D 246).[5]

(*e*) *Specific Gravity of Fractions.*—Standard Method of Test for Specific Gravity, 38/15.5 C., of Creosote Fractions (A.S.T.M. Designation: D 369).[5]

(*f*) *Float Test of Residue.*—The residue remaining in the retort after the distillation test shall be preserved until it reaches a temperature between 100 and 125 C. The consistency of the residue shall then be determined in accordance with the Standard Method of Float Test for Bituminous Materials (A.S.T.M. Designation: D 139).[5]

NOTE.—Care must be taken at the end of the distillation test to see that the vapor temperature as indicated on the thermometer does not rise above 355 C.; an excess temperature of only 1 or 2 C. at this point makes the float test invalid.

(*g*) *Coke Residue.*—Standard Method of Test for Coke Residue of Creosote (A.S.T.M. Designation: D 168).[5]

[5] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Method of Test for
COKE RESIDUE OF CREOSOTE[1]

A.S.T.M. Designation: D 168 - 30

ADOPTED, 1927; REVISED, 1930.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 168; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test covers the procedure for determining the coke residue (fixed carbon) in creosote or creosote - coal tar solution by coking the residue resulting from the distillation test and calculating the result based on the original oil.

Apparatus

2. (*a*) *Crucible.*—A platinum crucible shall be used, with tightly fitting cover of the inverted or capsule type having a depth of about 1 cm., provided with a hole 2 mm. in diameter at its center. The crucible shall have a capacity of 25 to 30 ml. and with cover shall weigh 25 to 30 g.

(*b*) *Furnace.*—A vertical electric tube furnace of the form shown in Fig. 1 or a Bunsen or Meker burner may be used.

Procedure

3. (*a*) The residue resulting from the distillation test, carried out in accordance with the Standard Method of Test for Distillation of Creosote (A.S.T.M. Designation: D 246) of the American Society for Testing Materials,[3] shall be poured directly into the tared crucible or into a tin box wherein it may be heated on a water or steam bath, but not over a flame. A 1 ± 0.1 g. portion of the residue shall be weighed into the the covered crucible.

(*b*) If the vertical electric tube furnace is used, the temperature of the furnace shall be controlled by a thermocouple at 950 ± 20 C. The crucible shall be suspended in the electric furnace for exactly 7 min., after which it shall be removed, cooled, and weighed.

(*c*) If a Bunsen or Meker burner is used, it shall have a free flame 20 cm. in height. The crucible shall set in a nichrome triangle with approximately two-thirds of its height below the triangle and with the bottom of the crucible 6 to 8 cm. above the top of the burner. Assurance of the desired temperature of 950 ± 20 C. may be indicated by the

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-7 on Timber.

[2] Prior to adoption as standard, this method was published as tentative from 1923 to 1927.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

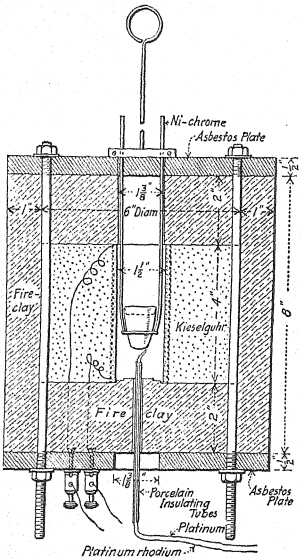


Fig. 1.—Electric Tube Furnace for Determining Coke Residue.

For 110-volt alternating current, 60 ft. of Nichrome Wire, No. 17 B. & S. gage will give the required temperature. The temperature must be controlled by an external resistance. (U. S. Bureau of Mines *Technical Paper No. 8*, p. 12 (1929)).

fusion of crystals of potassium chromate in the crucible when exposed in the specified position for the test. The crucible and contents shall be exposed to the full flame of the burner for exactly 7 min.

Precautions

4. The test shall be conducted in a part of the laboratory free from draughts.

Calculations

5. The percentage of coke obtained from the residue in accordance with Section 3 shall be calculated to percentage of total oil as follows:

$$\text{Coke in oil} = \frac{A \times B}{100}$$

where:

A = percentage residue from oil distilled to 355 C., and
B = percentage of coke in the residue.

NOTE: *Example.*—With a retort distillation of 29 per cent of residue at 355 C., the residue containing 28 per cent of fixed carbon:

$$\text{Coke in oil} = \frac{29 \times 28}{100} = 8.1 \text{ per cent}$$

Standard Method of Test for

DISTILLATION OF CREOSOTE[1]

A.S.T.M. Designation: D 246 – 42

ADOPTED, 1928; REVISED, 1930, 1933, 1939, 1942.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 246; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method covers a distillation test suitable for all types and grades of creosote, tar, and mixtures of creosote with tars used for timber preservation.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Flask.*—A side-neck distillation flask as shown in Fig. 1, having the following dimensions:

Diameter of bulb, outside	86 ± 1.5 mm.
Diameter of neck, inside	22 ± 1.0 mm.
Diameter of tubulature, inside	10 ± 0.5 mm.
Height of flask, outside	131 ± 1.5 mm.
Vertical distance bottom of bulb, outside, to horizontal tangent at tubulature inside	93 ± 1.5 mm.
Length of tubulature	220 ± 5.0 mm.
Angle of tubulature	73 ± 2 deg.
Thickness of tubulature wall	1.0 to 1.5

(*b*) *Condenser Tube.*—A suitable form of tapered glass condenser tube, having the following dimensions:

Diameter of small end	12.5 ± 1.5 mm.
Diameter of large end	28.5 ± 3.0 mm.
Length	360.0 ± 4.0 mm.
Length of tapered part	100.0 ± 5.0 mm.

(*c*) *Source of Heat.*—A source of heat consisting of a bunsen or equivalent burner or an electric heater. The electric heater,[3] of the form and dimensions shown in Fig. 2, shall be made of asbestos cement and shall consist of a base plate and cover interlocked to prevent side slipping, yet easily taken apart. The heating unit shall consist of an acid- and heat-resisting porcelain plate with coils of resistance wire of nickel-chromium alloy. It shall be a separate unit and readily renewable. The capacity of the heating unit shall be 600 watts and at full heat shall give a temperature of approximately 800 C. at the coils. Temperature of the heater shall be controlled by a sliding-contact tube rheostat of the tube rheostat laboratory type, which shall be approximately 41 mm. in diameter by 406 mm. in length. The resistance and continuous-ampere capacity of the rheostat shall be suitable for the voltage used.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-7 on Timber.

[2] Prior to adoption as standard, this method was published as tentative from 1926 to 1928, being revised in 1927 and 1928.

[3] The Gilmer electric heater controlled by the sliding-contact tube rheostat has been found entirely satisfactory For use with 110-v. heaters, a 14-ohm, 6.5-amp. rheostat should be used, and with 220-v. heaters, a 25-ohm, 5-amp. rheostat should be used.

(*d*) *Shield for Flame Distillation.*—A galvanized iron shield, lined with $\frac{1}{8}$-in. asbestos, of the form and dimensions shown in Fig. 3, used to protect the flask from air currents and to prevent radiation. If desired, the shield may be provided with a mica window to permit observation of the progress of distillation. The cover shall be made of transite board and shall be in two parts conforming to the dimensions shown in Fig. 3.

(*e*) *Shield for Electric-Heater Distillation.*—A galvanized-iron double-wall shield of the form and dimensions shown in Fig. 4, used to protect the flask from air currents and to prevent radiation. The space between the walls shall be filled with 85 per cent magnesia or powdered asbestos. If desired, the shield may be provided with a mica window to permit observation of the progress of distillation. The cover shall be made of transite board and shall be in two parts conforming to the dimensions shown in Fig. 4.

(*f*) *Receivers.*—Tared Erlenmeyer flasks having a capacity of 50 or 125 ml. for collecting the distillate.

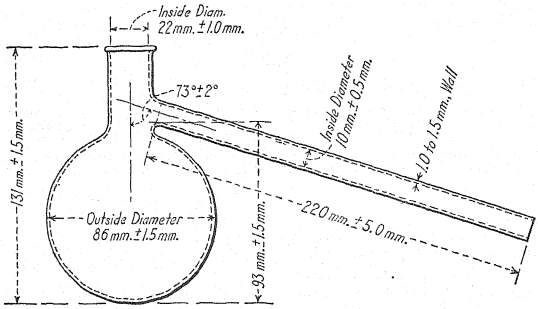


FIG. 1.—Distillation Flask.

(*g*) *Balance.*—A balance accurate to at least 0.05 g. for weighing the receivers and fractions.

(*h*) *Thermometer.*—An A.S.T.M. High Distillation Thermometer, total immersion, graduated in Centigrade degrees, having a range of 0 to 400 C., and conforming to the requirements for this thermometer as prescribed in the Standard Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1) of the American Society for Testing Materials.[4]

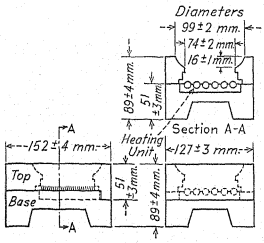


FIG. 2.—Electric Heater.

[4] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

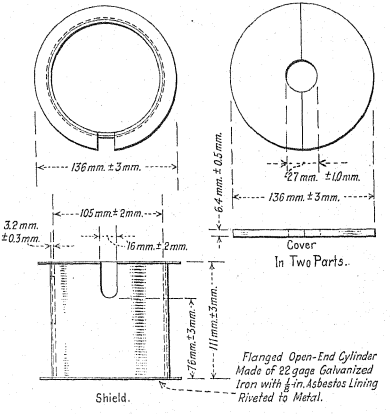


FIG. 3.—Shield and Cover for Flame Distillation.

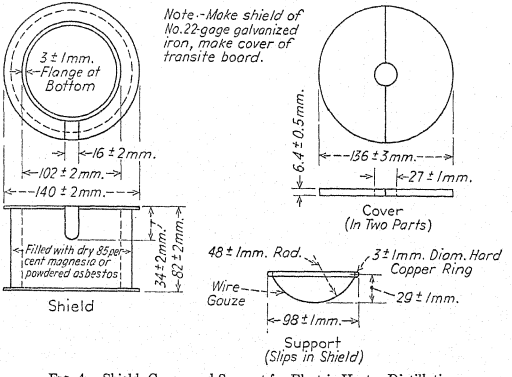


FIG. 4.—Shield, Cover, and Support for Electric-Heater Distillation.

Preparation of Sample

3. The sample as received should be thoroughly stirred and agitated, warming if necessary to insure a complete mixture free from crystallized solids, before the portion for analysis is removed.

Dehydration of Sample

4. If the presence of 3 per cent o water is suspected or known, the oi

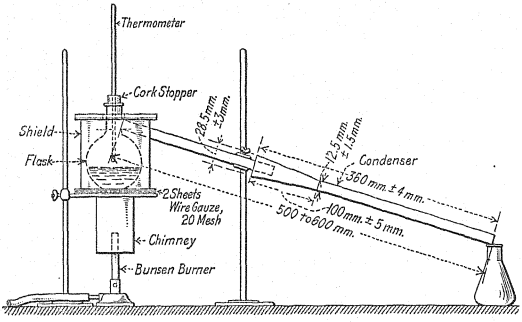


FIG. 5.—Apparatus Assembly for Flame Distillation.

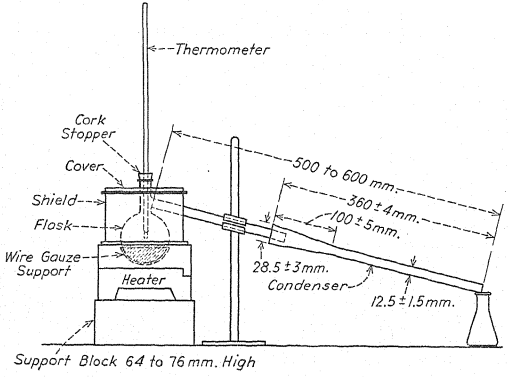


FIG. 6.—Apparatus Assembly for Electric-Heater Distillation.

shall be dehydrated before distillation in accordance with the Standard Method of Test for Water in Creosote (A.S.T.M. Designation: D 370) of the American Society for Testing Materials.[4]

Apparatus Assembly

5. (*a*) The flask shall be supported on a tripod or rings over two sheets of 20-mesh wire gauze, 150 mm. square, as shown in Fig. 5, or mounted on the

electric heater as shown in Fig. 6. The flask shall be connected to the condenser tube by a tight cork joint. The thermometer shall be inserted through a cork in the neck with the bottom of the bulb 12 to 13 mm. from the surface of the oil in the flask. The exact location of the thermometer bulb may be determined by calculating the number of divisions on the thermometer which are equal to 12 to 13 mm., lowering the tip of the bulb of the thermometer to the surface of the liquid and then raising the thermometer the calculated number of degrees to give the correct distance from the surface of the liquid.

(*b*) The axis of the bulb of the flask through the center of the neck shall be vertical and the thermometer shall be aligned on this axis.

(*c*) The distance from the neck of the flask to the outlet end of the condenser tube shall be not more than 600 nor less than 500 mm. The burner shall be protected from drafts by a suitable shield or chimney, see Fig. 5.

Procedure

6. (*a*) One hundred grams plus or minus 0.1 g. of the sample shall be weighed into the flask, the apparatus assembled, and heat applied so that the first drop of the distillate falls from the end of the condenser in 10 min. ± 100 sec.[5] The rate at the end of the condenser shall be adjusted to 90 to 100 drops per min. within 2 min. after the first drop and shall be maintained at 80 to 100 drops per min. throughout the distillation. The distillate shall be collected in weighed receivers. The condenser tube shall be warmed whenever necessary to prevent accumulation of solid distillates. Fractions shall be collected at the temperatures designated by the specifications. The receivers shall be changed as the mercury passes the dividing temperature for each fraction. When the maximum specified temperature of the test is indicated by the thermometer, heating shall be discontinued, the shield cover shall be removed immediately, and any distillate which has condensed in the condenser tube shall be drained into the last fraction. When the electric heater is used as a source of heat, the flask shall be removed from the assembly when the maximum specified temperature is indicated.

(*b*) The residue shall be retained in the flask with the cork and the thermometer in position until no vapors are visible; it shall then be weighed. The flask shall then be tipped around so that the residue will flow around the sides, thus collecting any condensed vapors that have collected on the sides of the flask, after which the portion for the coke residue determination may be withdrawn or the residue shall be poured into a suitable receptacle, and covered. If the residue becomes so cool that it cannot be poured readily from the flask, it shall be reheated to a temperature not exceeding 125 C. by holding the bulb of the flask in a suitable bath and not by the direct application of flame or heat.

(*c*) During the progress of the distillation the thermometer shall remain in its original position. No correction shall be made for the emergent stem of the thermometer.

Report

7. (*a*) The fractions obtained at the

[5] In cases of routine or plant control analyses where the presence of water is known or suspected, the sample shall be heated slowly until a temperature of 150 C. is reached. The heat shall then be increased and so regulated that the specified distillation rate will be reached in 5 min. From this point the method as specified shall be followed.

following temperatures shall be reported:

0 to 210 C.
210 to 235 C.
235 to 270 C.[a]
270 to 315 C.
315 to 355 C.
Residue.

[a] The fraction 235 to 270 C. is not usually required in specifications for creosote, but it is recommended that this cut be made, since it gives useful information.

(*b*) If the fraction obtained at 0 to 210 C. contains water, the amount of water shall be determined (Note). This amount shall then be deducted from the weight of oil taken and all of the fractions shall be corrected to a percentage based on the weight of the water-free sample.

NOTE.—A convenient method for determining the amount of water is to transfer this fraction, after weighing, to a tube or cylinder graduated in 0.1 ml. and add about 15 to 20 ml. of benzol. This almost always causes a clear separation between the oil and water.

Distillation Temperatures at Various Altitudes

8. If the elevation at which the distillation is to be made exceeds 1000 ft., the temperatures at which the fractions are taken shall be corrected in accordance with Table I.

TABLE I.—TEMPERATURES AT WHICH CREOSOTE FRACTIONS SHALL BE CUT TO CORRECT DISTILLATION TEMPERATURES FOR DIFFERENT ALTITUDES.

(Corrections made to the nearest degree Centigrade)

Elevation Above Sea Level, ft.	Fractionation Temperatures for Various Altitudes, deg. Cent.					
0.....	200	210	235	270	315	355
1000.....	198	208	233	268	313	353
1500.....	197	207	232	267	312	352
2000.....	196	206	231	266	311	351
2500.....	196	206	230	265	310	350
3000.....	195	205	230	264	309	349
3500.....	194	204	229	263	308	348
4000.....	193	203	228	263	307	347
4500.....	193	202	227	262	306	346
5000.....	192	202	226	261	305	344
5500.....	191	201	225	260	304	343
6000.....	190	200	225	260	303	343

Standard Methods of Test for

INSOLUBLE MATTER IN CREOSOTE[1]

A.S.T.M. Designation: D 367 – 33

ADOPTED, 1933.

This Standard of the American Society for Testing Materials is issued under the fixed designation D 367; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods are intended for the determination of insoluble matter (free carbon) in creosote and creosote - coal tar solutions by extraction with hot benzol. The porous thimble method is the preferred procedure and must be used in any case of dispute. The asbestos mat method is a routine procedure which gives equally accurate results and requires 3 or 4 hr. less time than the porous thimble extraction.

POROUS THIMBLE METHOD

Apparatus

2. (*a*) *Extractor.*—The extractor shall be of the form shown in Fig. 1 or of any similar form in which the oil is subjected to direct washing by the vapors of the boiling solvent.

(*b*) *Filter.*—The filtering medium shall be a flat bottom, 30 by 80 mm. RA 98 alundum thimble. The thimble shall either be suspended in the extraction flask by a wire basket hung from two small hooks on the under surface of the metal cover of the flask or it shall be supported by making perforations near the upper edge of the thimble and suspending from the cover by German silver or platinum wire.

(*c*) *Balance.*—The analytical balance shall be accurate to 0.5 mg.

Procedure

3. An amount of material which shall contain 10.0 ± 0.1 g. of dry oil shall be weighed into a 100-ml. beaker; 50 ml. of pure benzol shall be added and the solution stirred thoroughly. The solution shall be transferred at once to the weighed alundum thimble. The beaker shall be rinsed clean with pure benzol and the washings added to the thimble. The thimble shall then be covered with a lid of alundum ware and placed immediately in the extraction apparatus. The extractor shall contain a suitable quantity of pure benzol and shall be heated sufficiently to boil the solvent. The extraction shall be continued until the solvent descending from the thimble is colorless. The thimble shall then be dried at 105 ± 5 C., cooled in a desiccator, and weighed.

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-7 on Timber.

ASBESTOS MAT METHOD

Apparatus

4. (*a*) *Filter.*—The filtering medium shall consist of a No. 3 Gooch, Coors porcelain, or equivalent crucible approximately 3.5 cm. in diameter at the top, tapering to 2.2 cm. at the bottom

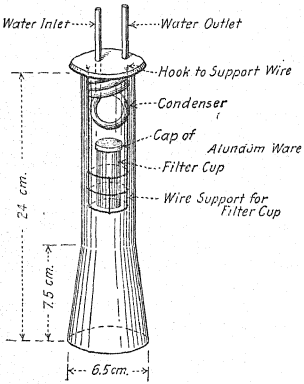


FIG. 1.—Extraction Flask.

with a depth of 4.0 cm., containing a mat of acid-washed medium fiber asbestos approximately 2 mm. in thickness.

(*b*) *Balance.*—The analytical balance shall be accurate to 0.5 mg.

Preparation of Filter Mat

5. The Gooch crucible shall be placed in the suction apparatus and filled with acid-washed medium fiber asbestos suspended in water. Gentle suction shall be applied and more of the suspension, if necessary, shall be added to make a mat approximately 2 mm. in thickness. With the suction still on, the pad shall be washed with water until all small particles of asbestos are removed. The crucible shall be dried at 105 ± 5 C., placed in a desiccator, and weighed.

Procedure

6. An amount of material which shall contain 10.0 ± 0.1 g. of dry oil shall be weighed into a 125-ml. Erlenmeyer flask; 50 ml. of pure benzol shall be added and the solution stirred thoroughly and brought to a boil. The hot solution shall be carefully poured into the weighed prepared Gooch crucible without suction until the mat is covered. Then gentle suction shall be applied to the crucible and the remaining solution added, taking care that the mat is covered with solution at all times. The Erlenmeyer flask shall be rinsed clean with pure benzol and the washings added to the crucible. Benzol shall be added to the crucible until the descending solvent is colorless. The crucible shall then be dried at 105 ± 5 C., cooled in a desiccator, and weighed.

Standard Method of Test for
SPECIFIC GRAVITY OF CREOSOTE[1]

A.S.T.M. Designation: D 368 – 33

ADOPTED, 1933.

This Standard of the American Society for Testing Materials is issued under the fixed designation D 368; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test covers the procedure for determining the specific gravity of creosote by means of the hydrometer.

Apparatus

2. (*a*) *Hydrometer.*—The hydrometer shall be of glass of the conventional constant mass and variable displacement type as shown in Fig. 1. A set of two hydrometers with ranges in specific gravity of from 1.000 to 1.080 and from 1.070 to 1.150 will suffice. The scale shall be divided into 0.001 of a unit of specific gravity. The hydrometer shall be standardized at 15.5/15.5 C. and shall conform to the following requirements as to dimensions:

	Dimension, mm.	Permissible Variation, mm.
Length of stem	125	±6
Length of bulb	105	±5
Length of scale	80	±4
Diameter of stem	6	±0.5
Diameter of bulb	22	±1

(*b*) *Hydrometer Cylinder.*—The hydrometer cylinder shall be of the form

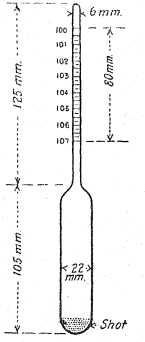


FIG. 1.—Hydrometer.

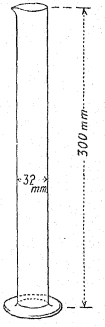


FIG. 2.—Hydrometer Cylinder.

shown in Fig. 2 and shall conform to the following requirements as to dimensions:

Length.....approximately 300 mm
Diameter, inside.....not less than 32 mm.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-7 on Timber.

Procedure

3. The sample of oil shall be brought to a temperature of 38 C. and the specific gravity shall be determined at that temperature unless the oil is not entirely liquid at 38 C. In case it is necessary to bring the oil to a temperature higher than 38 C. in order to render it completely fluid, the specific gravity shall be determined at the lowest temperature at which it is completely fluid. This observed specific gravity at the observed temperature shall be corrected to the specific gravity at 38 C. by the use of Table II of the Standard Volume and Specific Gravity Correction Tables for Creosote and Coal Tar (A.S.T.M. Designation: D 347) of the American Society for Testing Materials.[2]

[2] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Method of Test for

SPECIFIC GRAVITY, 38/15.5 C., OF CREOSOTE FRACTIONS[1]

A.S.T.M. Designation: D 369 - 33

ADOPTED, 1933.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 369; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method is a convenient and accurate means of determining the specific gravity of (*a*) creosote fractions (distilled at 235 to 315 C.) entirely liquid at 38 C., and (*b*) creosote fractions (distilled at 315 to 355 C.) containing solids at 38 C. It is also suitable for other distillation fractions of creosote or for creosote and creosote - coal tar solutions where only small amounts are available.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Pycnometers.*—Pycnometers of 10 and 25-ml. capacities with ground-glass stoppers having the usual capillary openings.

(*b*) *Water Bath.*—A water bath consisting of a vessel filled with sufficient water to permit of maintaining a temperature of 38 ± 0.1 C.

(*c*) *Thermometer.*—An A.S.T.M. Low Softening Point Thermometer graduated in Centigrade degrees, having a range of — 2 to + 80 C., and conforming to the requirements for this thermometer as prescribed in the Standard Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1) of the American Society for Testing Materials.[3]

(*d*) *Balance.*—An analytical balance accurate to 0.5 mg.

Calibration of Pycnometer

3. Before making a determination, the pycnometer with stopper shall first be calibrated as follows: Weigh the empty, clean, dry pycnometer with stopper upon the analytical balance. Fill the pycnometer with freshly boiled distilled water, place in the water bath, and bring the temperature to 38.0 ± 0.1 C. Insert stopper, wipe dry, and weigh.

Procedure for Fractions Entirely Liquid at 38 C.

4. The specific gravity of creosote fractions (distilled at 235 to 315 C.)

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-7 on Timber.

[2] Prior to adoption as standard, this method was published as tentative from 1930 to 1933, being revised in 1933.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

entirely liquid at 38 C. shall be determined as follows:

Heat the creosote fraction until completely liquid and continue heating to a temperature not exceeding 38 C.; then pour it into the empty, dry pycnometer until full, avoiding the formation of air bubbles. Insert the stopper in the pycnometer, taking precautions to avoid the inclusion of air bubbles. Place the filled pycnometer in the water bath maintained at a temperature of 38 ± 0.1 C. until the pycnometer and its contents are at a constant volume at 38 C. After immersion in the bath for at least 30 min., remove the pycnometer, wipe clean and dry; then weigh.

Procedure for Fractions Containing Solids at 38 C.

5. The specific gravity of creosote fractions (distilled at 315 to 355 C.) containing solids at 38 C. shall be determined as follows:

Heat the creosote fraction until completely liquid, and pour it into the empty, dry pycnometer until about one-half full, avoiding the inclusion of air bubbles. Cool to room temperature and weigh. Cover the solid or partially solid fraction with freshly boiled distilled water until the pycnometer is about three-quarters full; place in a water bath at 90 C. and allow to remain without agitation until the fraction is liquid and all air is removed. Cool the pycnometer and its contents to a temperature somewhat below 38 C.; fill the pycnometer with freshly boiled distilled water, avoiding the formation of air bubbles; and insert the stopper in the pycnometer, taking precautions to avoid the inclusion of air bubbles.

Place the filled pycnometer in the water bath maintained at a temperature of 38 ± 0.1 C. until the pycnometer and its contents are at a constant volume at 38 C. After immersion in the bath for at least 30 min., remove the pycnometer, wipe clean and dry; then weigh.

Calculations

6. The expression "38/15.5 C." means specific gravity of the fraction at 38 C. compared with water at 15.5 C. This cannot be determined directly. The specific gravity is first determined at 38 C. compared with water at 38 C. and this determination represents the relation of the weight of a volume of oil at 38 C. to the weight of an equal volume of water at the same temperature. The relation to an equal volume of water at 15.5 C. is obtained by multiplying the former figure by 0.99393—the density of water at 38 C. compared to water at 15.5 C., $\frac{(0.99299)}{(0.99905)}$. From the foregoing, it will be readily seen that it is incorrect to calculate the specific gravity at 38/15.5 C. by dividing the weight of the oil determined at 38 C. by the weight of water taken at 15.5 C.

NOTE: *Examples.*—The following examples are given to illustrate the correct methods of calculation:

Fractions Entirely Liquid at 38 C.—The weight of the pycnometer is 19.1624 g.; the weight of the pycnometer filled with water is 43.8837 g.; the weight of the pycnometer filled with oil is 44.876 g.:

$$\text{Sp. gr. at } 38/38 \text{ C.} = \frac{44.8676 - 19.1624}{43.8837 - 19.1624} = 1.0398$$

$$\text{Sp. gr. at } 38/15.5 \text{ C.} = 1.0398 \times 0.99393 = 1.0335$$

Fractions Containing Solids at 38 C.—The weight of the pycnometer is 12.6370 g.; the weight of the pycnometer filled with water is 22.5708 g.; the weight of the pycnometer partially filled with oil is 17.4962 g.; the weight of the partially filled pycnometer completely filled with water is 23.0453 g.:

$$\text{Sp. gr. at } 38/38\text{ C.} = \frac{17.4962 - 12.6370}{(22.5708 - 12.6370) - (23.0453 - 17.4962)} = 1.1082$$

$$\text{Sp. gr. at } 38/15.5 \text{ C.} = 1.1082 \times 0.99393 = 1.1015$$

Standard Method of Test for

WATER IN CREOSOTE[1]

A.S.T.M. Designation: D 370 – 33

ADOPTED, 1933.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 370; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This is an accurate method for determining the percentage of water in creosote and creosote - coal tar solution. Its use is indicated for dehydrating creosote containing more than 3 per cent of water in preparation for the distillation test. The alternative procedure is more convenient and rapid where the determination of water only is required.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Still.*—A vertical, cylindrical copper still, with removable flanged top and yoke, of the form and approximate dimensions shown in Fig. 1.

(*b*) *Thermometer.*—An A.S.T.M. High Distillation Thermometer, total immersion, graduated in either Centigrade or Fahrenheit degrees as specified, having a range of 0 to 400 C. or 30 to 760 F., and conforming to the requirements for this thermometer as prescribed in the Standard Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1) of the American Society for Testing Materials.[3]

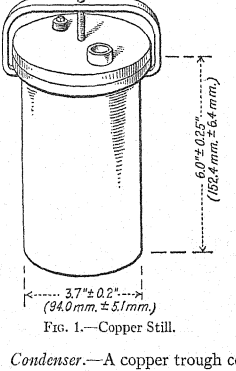


FIG. 1.—Copper Still.

(*c*) *Condenser.*—A copper trough condenser, with straight-walled glass tube, having approximately the form and dimensions shown in Fig. 2.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-7 on Timber.

[2] Editorially revised and rearranged in 1939.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(*d*) *Separatory Funnel.*—A separatory funnel conforming to the requirements shown in Fig. 3. It shall have a total capacity of approximately 200 ml. with the lower 20 ml. graduated accurately in 0.1 ml. The graduation marks shall be numbered for each milliliter.

Apparatus Assembly

3. The apparatus shall be assembled as shown in Fig. 2.

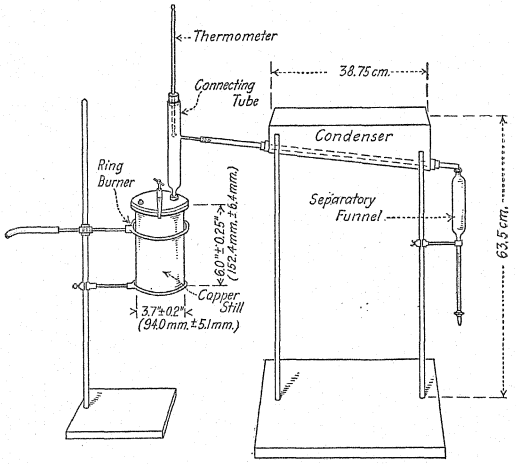


FIG. 2.—Assembled Apparatus for Water Test.

possible, and measured separately. If more than 3 per cent of water is present, the percentage of water present shall be determined by the method described in Paragraph (*b*), and the water-free oil so obtained shall be used in the distillation test.

(*b*) A 200-ml. sample of the oil shall be measured into a graduated cylinder and poured into a copper

Procedure

4. (*a*) When any measurable amount of water is present in the distillate below 210 C. as determined in accordance with the Standard Method of Test for Distillation of Creosote (A.S.T.M. Designation: D 246) of the American Society for Testing Materials,[3] the oil and water in this fraction shall be separated, if still, allowing the cylinder to drain into the still for several minutes. The lid shall be clamped on, using a paper gasket slightly wet with oil around the flange of the still. Heat shall be applied by means of the ring burner, which shall be placed just above the level of the oil in the still at the beginning of the test and gradually lowered when most of the water has distilled over. The dis-

tillation shall be continued until the vapor temperature reaches 205 C., as indicated by the thermometer with the bulb opposite the off-take of the connecting tube. The distillate shall be collected in a separatory funnel. When the distillation is completed, and a clear separation of water and oil in the funnel has taken place, the water shall be read by volume and drawn off. Any light oil distilled over with the water shall be returned to the oil in the still. The dehydrated oil from the still shall be used for the distillation test.

Alternative Procedure

5. The amount of water may be determined in accordance with the Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (A.S.T.M. Designation: D 95) of the American Society for Testing Materials.[3]

NOTE.—This method is of particular service where the determination of water only is involved and the dehydrated material is not to be used subsequently for analysis.

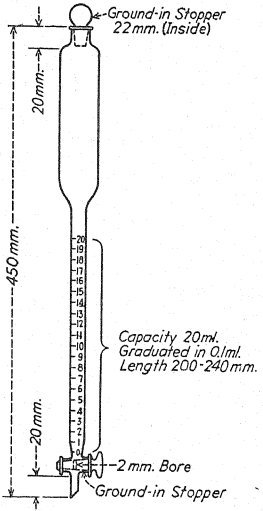


FIG. 3.—Glass Separatory Funnel, 200-Capacity.

Standard Method of Test for

TAR ACIDS IN CREOSOTE AND CREOSOTE - COAL TAR SOLUTIONS[1]

A.S.T.M. Designation: D 453 – 41

ADOPTED, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 453; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test covers the procedure for determining the amount of tar acids in fractions distilled from creosote and creosote - coal tar solutions.

Apparatus

2. (*a*) *Type I Separatory Funnel.*—The type I glass separatory funnel shall conform to the requirements shown in Fig. 1. It shall have a total capacity of approximately 200 ml. with the lower 20 ml. graduated accurately in 0.1 ml. The graduation marks shall be numbered for each milliliter.

(*b*) *Type II Separatory Funnel.*—The type II glass separatory funnel shall have a total capacity of approximately 260 ml. and shall conform to the requirements shown in Fig. 2. The capacity of the lower bulb from the stopcock to the first graduation mark shall be 65 ml., and above this mark the stem shall be graduated accurately for 100 ml. in 0.2 ml. The graduation marks shall be numbered for each two milliliters.

Procedure

3. (*a*) In making the determination, 100 g. of the sample shall be distilled in accordance with the Standard Method of Test for Distillation of Creosote (A.S.T.M. Designation: D 246) of the American Society for Testing Materials.[3] The fraction (Note) to be tested shall be transferred to a regular 250-ml. glass-stoppered separatory funnel, and 50 ml. of c.p. benzol and 50 ml. of an 18.3 per cent solution of NaOH (sp. gr. 1.20 at 20/4 C.) shall be added. The mixture shall be shaken vigorously for 3 min. and allowed to settle. The well-settled lower portion of the liquid shall then be drawn off into a 250-ml. beaker. An additional 30 ml. of the NaOH solution (18.3 per cent) shall be added to the separatory funnel and the contents gently shaken for 2 min. After settling, the lower portion of the liquid shall be drawn off and added to the beaker con-

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-7 on Timber.

[2] Prior to adoption as standard, this method was published as tentative from 1937 to 1941, being revised in 1938 and 1939.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

taining the first portion. Sufficient diluted H_2SO_4 (1:3) shall be added to the mixture in the beaker to turn blue litmus paper definitely red. The solution shall be cooled during the addition of the acid.

(*b*) If the tar acids liberated are estimated to be under 10 ml., type I separatory funnel (Fig. 1) shall be used and 10 ml. of high-flash naphtha or

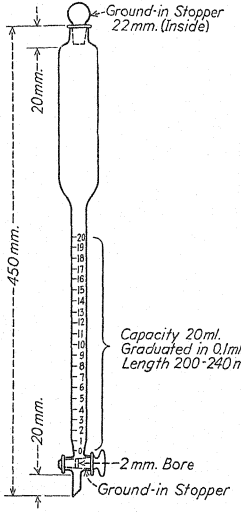


FIG. 1.—Type I Glass Separatory Funnel, 200-ml. Capacity.

c.p. benzol at 25 C. shall be measured into it. The liberated tar acids and sulfate solution shall then be poured through this layer of naphtha or benzol several times, drawing the material off at the bottom of the funnel into the original beaker and pouring it back into the top of the funnel. This washes out the beaker and allows all the tar acids to be absorbed. The funnel shall then be allowed to stand at 25 C., until the layers separate clearly, when the sulfate solution shall be drawn off and the increase in the volume of the naphtha or benzol taken as the dry tar acids present.

(*c*) If the tar acids liberated are estimated to be more than 10 ml., the procedure described in Paragraph (*b*) shall be followed except that 65 ml. of

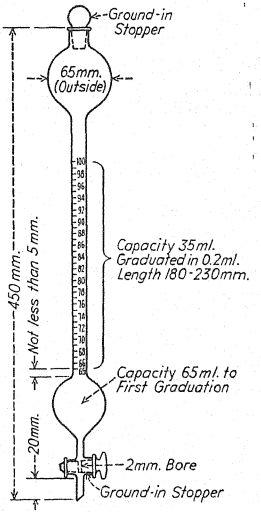


FIG. 2.—Type II Glass Separatory Funnel 260-ml. Capacity.

high-flash naphtha or c.p. benzol at 25 C. shall be measured into the type II separatory funnel (Fig. 2.).

NOTE.—If the total content of tar acids in a sample is desired, the entire distillate below 355 C. should be tested. In this case the results shall be reported as percentage by volume, that is, as the number of milliliters of tar acids per 100 ml. of the original dry sample, both volumes measured at the same temperature.

Calculation

4. The result shall be reported as a percentage of the fraction tested, calculated as follows:

$$\text{Tar acids, per cent} = \frac{G \times V}{W} \times 100$$

where G = specific gravity of the tar acids at 25 C./15.5 C.,[a]
V = volume of tar acids in milliliters, and
W = weight of fraction in grams.

[a] For practical purposes the specific gravity of the tar acids may be assumed to be 1.040.

STANDARD VOLUME AND SPECIFIC GRAVITY CORRECTION TABLES FOR CREOSOTE AND COAL TAR[1]

A.S.T.M. Designation: D 347 – 33

ADOPTED, 1933.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 347; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

These tables have been prepared by the National Bureau of Standards to meet a demand from the wood preserving industry for convenient tables for reducing creosote volumes to the basis of 100 F. (38 C.) and for reducing specific gravity observations to the same temperature. Tables I and II give in parallel columns correction factors for creosote, and for mixtures of creosote and coal tar (up to 50 per cent tar) designated as solution, and for coal tar. They are based on density determinations made on a selected range of domestic and foreign creosote and domestic coke-oven tars.

Table I shows the volume occupied at 100 F. (38 C.) by a quantity of oil occupying a unit volume at the indicated temperature; for example, 1 gal. of creosote measured at 120 F. (49 C.) will have a volume of 0.9921 gal. at 100 F. (38 C.); thus, if the volume of creosote at 120 F. (49 C.) equals 10,000 gal., then the volume at 100 F. (38 C.) equals 10,000 times 0.9921 or 9,921 gal.

Table II gives corrections for observed specific gravity which are simply made by adding them to the observed values for temperatures above 100 F. (38 C.) and subtracting them for temperatures below 100 F. (38 C.).

[1] Under the standardization procedure of the Society, these tables are under the jurisdiction of the A.S.T.M. Committee D-7 on Timber.

These tables have been adopted as standard by the American Wood Preservers' Association and by the American Railway Engineering Association; they are the outcome of a Joint Conference Committee representing these associations and the American Society for Testing Materials.

[2] Prior to adoption as standard, these tables were published as tentative from 1932 to 1933.

TABLE I.—VOLUME CORRECTION TABLE FOR CREOSOTE, CREOSOTE-COAL TAR SOLUTION (UP TO 50 PER CENT TAR), AND COAL TAR (COKE-OVEN TARS).

The observed volume is to be multiplied by the factor corresponding to the observed temperature.

Observed Temperature, deg. Fahr.	Volume at 100 F. Occupied by Unit Volume at Indicated Temperature			Observed Temperature, deg. Fahr.	Volume at 100 F. Occupied by Unit Volume at Indicated Temperature			Observed Temperature, deg. Fahr.	Volume at 100 F. Occupied by Unit Volume at Indicated Temperature		
	Creosote	Solution	Coal Tar		Creosote	Solution	Coal Tar		Creosote	Solution	Coal Tar
220	0.9526	0.9542	0.9594	180	0.9684	0.9696	0.9732	140	0.9842	0.9850	0.9867
219	0.9530	0.9546	0.9597	179	0.9687	0.9700	0.9735	139	0.9846	0.9853	0.9870
218	0.9534	0.9550	0.9600	178	0.9691	0.9704	0.9739	138	0.9850	0.9857	0.9874
217	0.9538	0.9554	0.9604	177	0.9695	0.9708	0.9742	137	0.9853	0.9861	0.9877
216	0.9542	0.9557	0.9607	176	0.9699	0.9712	0.9745	136	0.9857	0.9865	0.9880
215	0.9546	0.9561	0.9611	175	0.9703	0.9715	0.9749	135	0.9861	0.9868	0.9884
214	0.9550	0.9565	0.9614	174	0.9707	0.9719	0.9752	134	0.9865	0.9872	0.9887
213	0.9554	0.9569	0.9618	173	0.9711	0.9723	0.9756	133	0.9869	0.9876	0.9890
212	0.9558	0.9573	0.9621	172	0.9715	0.9727	0.9759	132	0.9873	0.9880	0.9894
211	0.9561	0.9577	0.9625	171	0.9719	0.9731	0.9762	131	0.9877	0.9884	0.9897
210	0.9565	0.9581	0.9628	170	0.9723	0.9735	0.9766	130	0.9881	0.9887	0.9900
209	0.9569	0.9584	0.9632	169	0.9727	0.9738	0.9769	129	0.9885	0.9891	0.9904
208	0.9573	0.9588	0.9635	168	0.9731	0.9742	0.9773	128	0.9889	0.9895	0.9907
207	0.9577	0.9592	0.9639	167	0.9735	0.9746	0.9776	127	0.9893	0.9899	0.9910
206	0.9581	0.9596	0.9642	166	0.9739	0.9750	0.9779	126	0.9897	0.9902	0.9914
205	0.9585	0.9600	0.9646	165	0.9743	0.9754	0.9783	125	0.9901	0.9906	0.9917
204	0.9589	0.9604	0.9649	164	0.9747	0.9758	0.9786	124	0.9905	0.9910	0.9920
203	0.9593	0.9608	0.9652	163	0.9751	0.9762	0.9789	123	0.9909	0.9914	0.9924
202	0.9597	0.9611	0.9656	162	0.9754	0.9765	0.9793	122	0.9913	0.9917	0.9927
201	0.9601	0.9615	0.9659	161	0.9758	0.9769	0.9796	121	0.9917	0.9921	0.9930
200	0.9605	0.9619	0.9663	160	0.9762	0.9773	0.9800	120	0.9921	0.9925	0.9934
199	0.9609	0.9623	0.9666	159	0.9766	0.9777	0.9803	119	0.9925	0.9929	0.9937
198	0.9612	0.9627	0.9670	158	0.9770	0.9781	0.9806	118	0.9929	0.9932	0.9940
197	0.9616	0.9631	0.9673	157	0.9774	0.9785	0.9810	117	0.9932	0.9936	0.9944
196	0.9620	0.9634	0.9677	156	0.9778	0.9788	0.9813	116	0.9936	0.9940	0.9947
195	0.9624	0.9638	0.9680	155	0.9782	0.9792	0.9816	115	0.9940	0.9944	0.9950
194	0.9628	0.9642	0.9684	154	0.9786	0.9796	0.9820	114	0.9944	0.9948	0.9954
193	0.9632	0.9646	0.9687	153	0.9790	0.9800	0.9823	113	0.9948	0.9951	0.9957
192	0.9636	0.9650	0.9690	152	0.9794	0.9804	0.9827	112	0.9952	0.9955	0.9960
191	0.9640	0.9654	0.9694	151	0.9798	0.9808	0.9830	111	0.9956	0.9959	0.9964
190	0.9644	0.9658	0.9697	150	0.9802	0.9811	0.9833	110	0.9960	0.9962	0.9967
189	0.9648	0.9662	0.9701	149	0.9806	0.9815	0.9837	109	0.9964	0.9966	0.9970
188	0.9652	0.9665	0.9704	148	0.9810	0.9819	0.9840	108	0.9968	0.9970	0.9974
187	0.9656	0.9669	0.9708	147	0.9814	0.9823	0.9844	107	0.9972	0.9974	0.9977
186	0.9660	0.9673	0.9711	146	0.9818	0.9827	0.9847	106	0.9976	0.9978	0.9980
185	0.9664	0.9677	0.9714	145	0.9822	0.9830	0.9850	105	0.9980	0.9981	0.9983
184	0.9668	0.9681	0.9718	144	0.9826	0.9834	0.9854	104	0.9984	0.9985	0.9987
183	0.9672	0.9685	0.9721	143	0.9830	0.9838	0.9857	103	0.9988	0.9989	0.9990
182	0.9676	0.9688	0.9725	142	0.9834	0.9842	0.9860	102	0.9992	0.9992	0.9993
181	0.9680	0.9692	0.9728	141	0.9838	0.9846	0.9864	101	0.9996	0.9996	0.9997
								100	1.0000	1.0000	1.0000

The portion of the table below should not be used unless the oil is entirely free from crystals.

99	1.0004	1.0004	1.0003	84	0.0063	0.0062	0.0053	69	1.0122	1.0116	1.0103
98	1.0008	1.0008	1.0007	83	0.0067	0.0066	0.0056	68	1.0126	1.0120	1.0106
97	1.0012	1.0011	1.0010	82	0.0071	0.0070	0.0060	67	1.0130	1.0124	1.0109
96	1.0016	1.0015	1.0013	81	0.0075	0.0074	0.0063	66	1.0134	1.0127	1.0112
95	1.0020	1.0019	1.0017	80	0.0079	0.0078	0.0066	65	1.0138	1.0131	1.0116
94	1.0024	1.0022	1.0020	79	1.0083	1.0079	1.0070	64	1.0142	1.0135	1.0119
93	1.0028	1.0026	1.0023	78	1.0087	1.0082	1.0073	63	1.0146	1.0138	1.0122
92	1.0032	1.0030	1.0026	77	1.0091	1.0086	1.0076	62	1.0150	1.0142	1.0126
91	1.0036	1.0034	1.0030	76	1.0095	1.0090	1.0080	61	1.0154	1.0146	1.0129
90	1.0040	1.0038	1.0033	75	1.0099	1.0094	1.0083	60	1.0158	1.0150	1.0132
89	1.0043	1.0041	1.0036	74	1.0103	1.0097	1.0086				
88	1.0047	1.0045	1.0040	73	1.0107	1.0101	1.0089				
87	1.0051	1.0049	1.0043	72	1.0111	1.0105	1.0093				
86	1.0055	1.0052	1.0046	71	1.0115	1.0109	1.0096				
85	1.0059	1.0056	1.0050	70	1.0118	1.0112	1.0099				

TABLE II.—SPECIFIC GRAVITY CORRECTION TABLE FOR CREOSOTE, CREOSOTE-COAL TAR SOLUTION (UP TO 50 PER CENT TAR), AND COAL TAR (COKE-OVEN TARS).

Observed Temperature, deg. Fahr.	Add to Observed Specific Gravity		
	Creosote	Solution	Coal Tar
220	0.0491	0.0481	0.0451
219	0.0487	0.0477	0.0447
218	0.0483	0.0473	0.0443
217	0.0479	0.0469	0.0439
216	0.0475	0.0465	0.0436
215	0.0471	0.0460	0.0432
214	0.0467	0.0456	0.0428
213	0.0462	0.0452	0.0424
212	0.0458	0.0448	0.0420
211	0.0454	0.0444	0.0417
210	0.0450	0.0440	0.0413
209	0.0446	0.0436	0.0409
208	0.0442	0.0432	0.0405
207	0.0438	0.0428	0.0401
206	0.0434	0.0424	0.0398
205	0.0430	0.0420	0.0394
204	0.0426	0.0416	0.0390
203	0.0422	0.0412	0.0386
202	0.0418	0.0408	0.0382
201	0.0413	0.0404	0.0379
200	0.0409	0.0400	0.0375
199	0.0405	0.0396	0.0371
198	0.0401	0.0392	0.0367
197	0.0397	0.0388	0.0363
196	0.0392	0.0384	0.0360
195	0.0388	0.0379	0.0356
194	0.0384	0.0375	0.0352
193	0.0380	0.0371	0.0348
192	0.0375	0.0367	0.0344
191	0.0371	0.0363	0.0341
190	0.0367	0.0359	0.0337
189	0.0363	0.0355	0.0333
188	0.0359	0.0351	0.0329
187	0.0355	0.0347	0.0325
186	0.0351	0.0343	0.0322
185	0.0347	0.0339	0.0318
184	0.0343	0.0335	0.0314
183	0.0339	0.0331	0.0310
182	0.0334	0.0327	0.0306
181	0.0330	0.0323	0.0303
180	0.0326	0.0319	0.0299
179	0.0322	0.0315	0.0295
178	0.0318	0.0311	0.0291
177	0.0314	0.0307	0.0288
176	0.0309	0.0303	0.0284
175	0.0305	0.0298	0.0280
174	0.0301	0.0294	0.0276
173	0.0297	0.0290	0.0272
172	0.0293	0.0286	0.0269
171	0.0289	0.0282	0.0265
170	0.0284	0.0278	0.0261
169	0.0280	0.0274	0.0257
168	0.0276	0.0270	0.0254
167	0.0272	0.0266	0.0250
166	0.0268	0.0262	0.0246
165	0.0264	0.0258	0.0242
164	0.0260	0.0254	0.0238
163	0.0256	0.0250	0.0234
162	0.0252	0.0246	0.0231
161	0.0248	0.0242	0.0227
160	0.0244	0.0238	0.0223
159	0.0239	0.0234	0.0219
158	0.0234	0.0230	0.0216
157	0.0230	0.0226	0.0212
156	0.0226	0.0222	0.0208
155	0.0222	0.0218	0.0204
154	0.0218	0.0214	0.0200
153	0.0214	0.0210	0.0197
152	0.0210	0.0206	0.0193
151	0.0205	0.0202	0.0189
150	0.0202	0.0198	0.0186
149	0.0198	0.0194	0.0182
148	0.0194	0.0190	0.0178
147	0.0189	0.0186	0.0174
146	0.0186	0.0183	0.0170
145	0.0182	0.0178	0.0167
144	0.0177	0.0174	0.0163
143	0.0173	0.0170	0.0159
142	0.0169	0.0166	0.0155
141	0.0165	0.0162	0.0152
140	0.0161	0.0158	0.0148
139	0.0157	0.0154	0.0144
138	0.0153	0.0150	0.0141
137	0.0149	0.0146	0.0137
136	0.0145	0.0142	0.0133
135	0.0141	0.0138	0.0130
134	0.0137	0.0134	0.0126
133	0.0133	0.0130	0.0122
132	0.0129	0.0126	0.0118
131	0.0125	0.0122	0.0115
130	0.0121	0.0118	0.0111
129	0.0117	0.0114	0.0107
128	0.0113	0.0110	0.0104
127	0.0109	0.0106	0.0100
126	0.0105	0.0102	0.0096
125	0.0101	0.0098	0.0092
124	0.0097	0.0094	0.0089
123	0.0093	0.0090	0.0085
122	0.0089	0.0087	0.0081
121	0.0084	0.0083	0.0078
120	0.0080	0.0079	0.0074
119	0.0076	0.0075	0.0070
118	0.0072	0.0071	0.0067
117	0.0068	0.0067	0.0063
116	0.0064	0.0063	0.0059
115	0.0060	0.0059	0.0056
114	0.0056	0.0055	0.0052
113	0.0052	0.0051	0.0048
112	0.0048	0.0047	0.0044
111	0.0044	0.0043	0.0041
110	0.0040	0.0039	0.0037
109	0.0036	0.0035	0.0033
108	0.0032	0.0031	0.0030
107	0.0028	0.0027	0.0026
106	0.0024	0.0023	0.0022
105	0.0020	0.0020	0.0018
104	0.0016	0.0016	0.0015
103	0.0012	0.0012	0.0011
102	0.0008	0.0008	0.0007
101	0.0004	0.0004	0.0004
100	0.0000	0.0000	0.0000

The portion of the table below should not be used unless the oil is entirely free from crystals.

Observed Temperature, deg. Fahr.	Subtract from Observed Specific Gravity		
	Creosote	Solution	Coal Tar
99	0.0004	0.0004	0.0004
98	0.0008	0.0008	0.0007
97	0.0012	0.0012	0.0011
96	0.0016	0.0016	0.0015
95	0.0020	0.0020	0.0018
94	0.0024	0.0023	0.0022
93	0.0028	0.0027	0.0026
92	0.0032	0.0031	0.0030
91	0.0036	0.0035	0.0033
90	0.0040	0.0039	0.0037
89	0.0043	0.0043	0.0040
88	0.0047	0.0047	0.0044
87	0.0051	0.0051	0.0048
86	0.0055	0.0055	0.0052
85	0.0059	0.0058	0.0055
84	0.0063	0.0062	0.0059
83	0.0067	0.0066	0.0063
82	0.0071	0.0070	0.0066
81	0.0075	0.0074	0.0070
80	0.0079	0.0078	0.0074
79	0.0083	0.0082	0.0077
78	0.0087	0.0086	0.0081
77	0.0091	0.0090	0.0085
76	0.0095	0.0094	0.0088
75	0.0099	0.0097	0.0092
74	0.0103	0.0101	0.0096
73	0.0107	0.0105	0.0099
72	0.0111	0.0109	0.0102
71	0.0115	0.0113	0.0106
70	0.0118	0.0117	0.0109
69	0.0122	0.0121	0.0113
68	0.0126	0.0124	0.0117
67	0.0130	0.0128	0.0120
66	0.0134	0.0132	0.0124
65	0.0138	0.0136	0.0128
64	0.0142	0.0140	0.0132
63	0.0146	0.0144	0.0135
62	0.0150	0.0148	0.0139
61	0.0154	0.0152	0.0143
60	0.0158	0.0155	0.0146

Standard Methods of
CHEMICAL ANALYSIS OF ZINC CHLORIDE[1]

A.S.T.M. Designation: D 199 - 27

ADOPTED, 1927.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 199; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods cover the determination of the percentages of insoluble basic zinc chloride, zinc chloride, and iron present in commercial concentrated solutions of zinc chloride or in fused or granulated zinc chloride, for use in the preservative treatment of wood.

Special Solutions Required

2. (*a*) *Standard Ferrocyanide Solution.*—Weigh 43.25 g. of c.p. potassium ferrocyanide and 14 g. of c.p. crystallized sodium sulfite, dissolve in water and make up to 1 liter at room temperature. Shake thoroughly. Standardize against a zinc solution of known concentration prepared from spelter of known zinc content or from c.p. zinc oxide which has been previously ignited. One milliliter of this solution will be equal to approximately 0.01 g. of zinc. The standardization should be carried out as nearly as possible in the same manner as in the estimation of zinc and approximately the same amount of zinc should be present. Keep the solution in a dark bottle. Shake thoroughly before each using and standardize each time it is used.

(*b*) *Uranium Acetate Indicator.*—Dissolve 4.4 g. of c.p. uranium acetate, free from sodium, in 100 ml. of hot water and 2 ml. of glacial acetic acid. Use this as an external indicator on a paraffined plate making the drops as nearly 0.05 ml. as possible.

(*c*) *Hydrogen Peroxide.*—The usual laboratory reagent is satisfactory, if fresh.

(*d*) *Hydrogen Sulfide Solution.*—The usual laboratory reagent is satisfactory.

Insoluble or Basic Zinc Chloride

3. Weigh from 10 to 14 g. of the sample, if fused or granulated, from a stoppered weighing bottle, or place an equivalent quantity, if a solution, into a 600-ml. beaker. Add cold water to 400 ml. Stir the contents of the beaker until solution is complete. Allow to settle over night. Filter the solution through a 12.5-cm. filter paper which has pre-

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-7 on Timber.

[2] Prior to adoption as standard, these methods were published as tentative from 1924 to 1927. Editorially revised and rearranged in 1939.

viously been washed, dried, and weighed. Receive the filtrate in a 1000-ml. graduated measuring flask. Clean the beaker with a rubber policeman, and wash the insoluble matter in the filter paper until the filtrate measures 1 liter. Dry the filter paper containing the insoluble matter over night in an oven heated to 100 C. (212 F.). Cool, and weigh between clipped watch glasses. Calculate the increase in weight of filter paper to a percentage of the original sample.[3] Duplicate determinations should check within 1.3 per cent.

Zinc

4. (*a*) *Volumetric Method* (For use when manganese chloride does not exceed 0.3 per cent.).—Shake the filtrate obtained from the estimation of insoluble basic zinc chloride and whose volume is exactly 1 liter (Section 3), and transfer three aliquot portions of 100 ml. each with an accurate 100-ml. pipette into 450-ml. Griffin beakers. Add to each portion 15 g. of ammonium chloride and 5 ml. of HCl (sp. gr. 1.19). Dilute to 350 ml. and heat nearly to boiling. Titrate slowly, while stirring vigorously, using a solution of potassium ferrocyanide as the standard reagent and uranium acetate as an external indicator. Report the average of the results obtained with the three aliquot portions analyzed.

(*b*) *Volumetric Method* (For use when the manganese chloride equals or exceeds 0.3 per cent.).—To the aliquot portions taken as described in Paragraph (*a*), add 1 ml. of H_2O_2 (2 to 3 per cent) and 10 ml. of NH_4OH (1:1). Stand on steam bath until settled. Filter off the manganese, wash the beaker and paper twice with hot water. Dissolve the precipitate in the smallest amount of HCl (1:1) in the original beaker, heat until all is dissolved; the volume of the solution should be about 20 ml. Reprecipitate the manganese with 1 ml. of H_2O_2 and 10 ml. of NH_4OH, boil, filter, and wash several times with hot water. Add the filtrate to that obtained in the first separation. Add 15 ml. of HCl (sp. gr. 1.19) to the combined filtrate and just neutralize with NH_4OH (sp. gr. 0 90), then add 5 ml. of HCl in excess. Dilute to 325 ml. and add 25 ml. of saturated hydrogen sulfide water to remove any traces of H_2O_2, heat and titrate as described in Paragraph (*a*).

(*c*) *Gravimetric Method* (Alternate method, for use when only a limited amount of work is necessary.).—Shake the filtrate obtained from the estimation of insoluble basic zinc chloride and whose volume is exactly 1 liter (Section 3), and transfer three aliquot portions of 200 ml. each with an accurate pipette to 450-ml. Griffin beakers. Add to each 4 ml. of c.p. H_2SO_4 (sp. gr. 1.84). Evaporate on a steam bath; then on a steam plate or hot plate to copious SO_3 fumes, to completely eliminate chlorides. Cool, and take up in 100 ml. of hot distilled water. Add 0.5 g. of aluminum powder. Cover with a watch glass. Heat to boiling and boil 5 min. Filter through an 11-cm. filter paper. Receive the filtrate in a covered 1000-ml. Griffin beaker. Wash the beaker and filter thoroughly with hot water until a drop of methyl orange indicator placed behind the double fold of filter paper shows no acidity. Exactly neutralize the filtrate with diluted NH_4OH. Use great care and precision, and carry the neutralization just to the end point.

Add 10 ml. of 0.1 *N* H_2SO_4 (3 ml. of c.p. H_2SO_4 (sp. gr. 1.84) in 1000 ml. of water). Dilute to 650 ml. Cover the beaker, and bubble through the solution a rapid stream of H_2S for 30 min. to 1 hr. at room temperature.

Settle and filter through a double

[3] A Gooch crucible may be used in place of the weighed filter paper.

filter of one 15-cm. and one 11-cm. paper[4] folded together. Transfer the precipitate to the filter paper. Thoroughly clean the beaker with a rubber policeman until the zinc sulfide is all removed except a very thin film which clings tenaciously to the glass at the surface of the liquid. Thoroughly wash the beaker and precipitate at room temperature with water saturated with hydrogen sulfide. Repeat washing of filter paper, and precipitate five or six times. Transfer paper and precipitate to an ignited, cooled, desiccated, and weighed porcelain crucible of suitable capacity (about 25 to 30 ml.). Carefully dry the paper and precipitate and when dry completely burn off the paper at as low a temperature as possible. When carbon has been completely burned out, ignite the resultant oxide of zinc strongly to as high a temperature as is available, but not higher than can be attained with a laboratory blast lamp with the aid of gas and air. Heat for 30 min. After strong ignition, cool the crucible, desiccate, and weigh. The increase in weight is zinc oxide. The weight of the zinc oxide multiplied by the factor 1.6749, multiplied by 100, divided by the weight of sample in the aliquot portion taken, equals the percentages of zinc chloride.

Report the average of the results obtained with the three aliquot portions analyzed. Results should agree within 1.5 per cent.

Estimation of Iron and Aluminum

5. Weigh 10 g. of the sample, if fused or granulated, or place an equivalent quantity, if a solution, into a suitable beaker and dissolve in 100 ml. of water or dilute to 100 ml., if a solution. Add sufficient HCl to dissolve any basic zinc chloride. Add a slight excess of bromine water and boil off excess. Neutralize with a weak solution of sodium carbonate until a permanent precipitate of zinc carbonate is obtained. Add three drops of glacial acetic acid and 2 g. of sodium acetate, and boil. Filter and wash. Redissolve the precipitate in the original beaker with hot HCl (1:1). Reprecipitate the iron and alumina with a slight excess of NH_4OH, filter, and wash free from chlorine. Ignite in a platinum crucible and weigh as $Fe_2O_3 + Al_2O_3$.

[4] Ashless paper should be used.

Standard Definitions of

TERMS RELATING TO TIMBER PRESERVATIVES[1]

A.S.T.M. Designation: D 324 – 41

ADOPTED, 1933; REVISED, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 324; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Coal Tar.—Tar produced by the destructive distillation of bituminous coal.

Coke-Oven Tar.—Coal tar produced in by-product coke ovens in the manufacture of coke from bituminous coal.

Creosote (Creosote Oil, Creosote Distillate).— As used in wood preservation, a distillate of coal tar produced by high-temperature carbonization of bituminous coal; it consists principally of liquid and solid aromatic hydrocarbons, and contains appreciable quantities of tar acids and tar bases; it is heavier than water; and has a continuous boiling range of at least 125 C. beginning at about 200 C.

Creosote - Coal Tar Solution.—Solution of coal tar in creosote in various proportions. Usually contains 20 to 40 per cent of coal tar.

Creosote Distillate.—See Creosote.

Creosote Oil.—See Creosote.

Gas-House Coal Tar.—Coal tar produced in gas-house retorts in the manufacture of illuminating gas from bituminous coal.

Water-Gas Tar.—Tar produced in the manufacture of carbureted water gas by the decomposition of petroleum oil by heat in the presence of blue gas.

[1] Under the standardization procedure of the Society, these definitions are under the jurisdiction of the A.S.T.M. Committee D-7 on Timber.

[2] Prior to adoption as standard, these definitions were published as tentative from 1930 to 1933, being revised in 1933.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: K22.1-1944

Standard Specifications for

ZINC OXIDE[1]

A.S.T.M. Designation: D 79 - 44

ADOPTED, 1924; REVISED, 1939, 1941, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 79; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover the pigments commercially known as "zinc white" or zinc oxide. The pigment may be purchased in the dry form or as a paste in oil.

Composition and Properties

2. (*a*) *Dry Pigment.*—The pigment shall be zinc oxide of either the American process type or the French process type. The type desired shall be stated in the order. The pigment shall conform to the following requirements:

	American Process Type	French Process Type
Zinc oxide, min., per cent	98	99
Total sulfur, max., per cent	0.2	0.1
Moisture and other volatile matter, max., per cent	0.5	0.5
Total impurities, including moisture and other volatile matter, max., per cent	2.0	1.0
Coarse particles (total residue retained on a No. 325 (44-micron) sieve), max., per cent	1.0	1.0

(*b*) *Paste in Oil.*—The paste shall be made by thoroughly grinding the specified pigment with linseed oil. As received it shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. The paste shall conform to the following requirements:

Pigment, per cent	80 to 86
Linseed oil, per cent	14 to 20
Moisture and other volatile matter, max., per cent	0.5
Coarse particles and skins (total residue retained on a No. 325 (44-micron) sieve), max., per cent of the dry pigment	1.5

(*c*) In such physical properties as are specified by the purchaser, the pigment shall satisfactorily match a reference sample mutually agreed upon by the purchaser and the seller. In the event that either an acicular type or a nodular (spherical) type of zinc oxide is desired, the particle shape shall be determined by examining or photographing microscopic mounts (1000× or more) of the sample and the mutually agreed upon standard which are to be prepared as specified in the Tentative Method of Test for Par-

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1921 to 1924, being revised in 1924.

ticle Size Distribution of Subsieve Size Particulate Substances (A.S.T.M. Designation: E 20).[3]

Number of Tests

3. Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10,000 lb., except that for shipments of less than 10,000 lb. two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

Methods of Testing

4. Tests shall be conducted in accordance with the appropriate methods of the American Society for Testing Materials, where applicable. Test procedures not covered by A.S.T.M. methods shall be mutually agreed upon by the purchaser and the seller.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for

LEADED ZINC OXIDE[1]

A.S.T.M. Designation: D 80 – 41

ADOPTED, 1924; REVISED, 1940, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 80; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover the pigment commercially known as leaded zinc oxide. The pigment may be purchased in the dry form or as a paste in oil.

Composition and Properties

2. (*a*) *Dry Pigment.*—The pigment shall consist of zinc oxide and normal or basic lead sulfate, and shall conform to the following requirements:

	35 per cent Leaded
Zinc oxide, per cent	62 to 67
Matter soluble in water, max., per cent	1.0[a]
Moisture and other volatile matter, max., per cent	0.5[a]
Total impurities, including moisture and other volatile matter, max., per cent	2.0[a]
Normal or basic lead sulfate	remainder
Coarse particles (total residue retained on a No. 325 (44-micron) sieve), max., per cent	1.0[a]

[a] These specification limits and the permissible variation for zinc oxide content (plus 2 and minus 3 per cent, based on the percentage of zinc oxide claimed) apply to any other combination of zinc oxide and normal or basic lead sulfate that is agreed upon by the purchaser and the seller.

(*b*) *Paste in Oil.*—The paste shall be a dispersion of the specified pigment in linseed oil. As received it shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions, without curdling, with linseed oil, turpentine, or volatile petroleum spirits, or any mixture of these substances. The paste shall conform to the following requirements:

	35 per cent Leaded[a]
Pigment, per cent	81 to 87
Linseed oil, per cent	13 to 19
Moisture and other volatile matter, max., per cent	0.5
Coarse particles and skins (total residue retained on a No. 325 (44-micron) sieve), max., per cent of the dry pigment	1.5

[a] These specification limits may also apply to any other combination of zinc oxide and normal or basic lead sulfate that is agreed upon by the purchaser and the seller.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1921 to 1924, being revised in 1924.

(*c*) In such physical properties as are specified by the purchaser, the pigment shall satisfactorily match a reference sample mutually agreed upon by the purchaser and the seller.

Number of Tests

3. Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10,000 lb., except that for shipments of less than 10,000 lb. two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

Methods of Testing

4. Tests shall be conducted in accordance with the appropriate methods of the American Society for Testing Materials, where applicable. Test procedures not covered by A.S.T.M. methods shall be mutually agreed upon by the purchaser and the seller.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: K23.1-1943

Standard Specifications for

BASIC CARBONATE WHITE LEAD[1]

A.S.T.M. Designation: D 81 – 43

ADOPTED, 1924; REVISED, 1931, 1934, 1938, 1941, 1943.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 81; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover the material commercially known as basic carbonate white lead, used as a pigment and in putty. The pigment may be purchased in the dry form or as a paste in oil.

Composition and Properties

2. (*a*) *Dry Pigment.*—The pigment shall be free from adulterants and shall contain not more than traces of impurities incident to well-controlled manufacture of high-grade basic carbonate white lead. The pigment shall conform to the following requirements:

Lead carbonate, per cent	62.0 to 75.0
Moisture and other volatile matter, max., per cent	0.7
Total other impurities, max., per cent	1.0
Coarse particles (total residue retained on a No. 325 (44-micron) sieve), max., per cent	1.0

(*b*) *Paste in Oil.*—The paste shall be made by thoroughly grinding the specified pigment with linseed oil. The paste shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. The paste shall conform to the following requirements:

Pigment, min., per cent	89.0
Linseed oil, max., per cent	11
Moisture and other volatile matter, max., per cent	0.7
Coarse particles and skins (total residue retained on a No. 325 (44-micron) sieve), max., per cent of the dry pigment	1.5

(*c*) *Semipaste Containing Volatile Thinner.*—The semipaste shall be made by thoroughly grinding the specified pigment with a mixture of linseed oil and a small amount of volatile thinner. The semipaste shall not be caked in the container and shall be readily stirred to a uniform mixture which shall mix readily with oil, turpentine, or volatile petroleum spirits to form a smooth paint of brushing consistency. The odor of the semipaste, as taken from the container, while drying or after drying, shall be not abnormally pungent or disagreeable. The semi-

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1921 to 1924, being revised in 1924.

paste shall conform to the following requirements:

Pigment, min., per cent	87.5
Linseed oil, max., per cent	10.5
Moisture and other volatile matter[a], per cent	1.5 to 3.0
Moisture, max., per cent	0.7
Coarse particles and skins (total residue retained on a No. 325 (44-micron) sieve), max., per cent of the dry pigment	1.5

[a] The volatile matter shall be turpentine, volatile petroleum spirits, or any mixture thereof.

(*d*) The color and color strength, when specified, shall be equal to that of a reference sample mutually agreed upon by the purchaser and the seller.

Number of Tests

3. Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10,000 lbs., except that for shipments of less than 10,000 lb. two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

Methods of Testing

4. Tests shall be conducted in accordance with the appropriate methods of the American Society for Testing Materials, where applicable. Test procedures not covered by A.S.T.M. methods shall be mutually agreed upon by the purchaser and the seller.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
A.S.A. NO.: K47.1-1944

Standard Specifications for

BASIC SULFATE WHITE LEAD

A.S.T.M. Designation: D 82 – 4[1]

ADOPTED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 82; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover the pigment commercially known as basic sulfate white lead. The pigment may be purchased in the dry form or as a paste in oil.

Composition and Properties

2. (*a*) *Dry Pigment.*—The pigment shall conform to the following requirements:

Lead oxide, per cent	15.0 to 28.0
Zinc oxide, max., per cent	5.0
Total impurities, including moisture and other volatile matter, max., per cent	1.0
Lead sulfate	remainder
Coarse particles (total residue retained on a No. 325 (44-micron) sieve), max., per cent.	1.0

(*b*) *Paste in Oil.*—The paste shall be made by thoroughly grinding the specified pigment with linseed oil. The paste as received shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions, without curdling, with linseed oil, turpentine, or volatile petroleum spirits, or any mixture of these substances. The paste shall conform to the following requirements:

Pigment, min., per cent	84.0
Linseed oil, max., per cent	16.0
Moisture and other volatile matter, max., per cent	0.7
Coarse particles and skins (total residue retained on a No. 325 (44-micron) sieve), max., per cent of the dry pigment	1.5

(*c*) The color and color strength, when specified, shall be equal to that of a reference sample mutually agreed upon by the purchaser and the seller.

Number of Tests

3. Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1921 to 1924, being revised in 1924. They were adopted in 1924, published as standard from 1924 to 1942, being revised in 1938 and 1941, but withdrawn, revised, and republished as tentative in 1942.

ratio of two samples for each 10,000 lb., except that for shipments of less than 10,000 lb. two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

Methods of Test

4. Tests shall be conducted in accordance with the appropriate methods of the American Society for Testing Materials, where applicable. Test procedures not covered by A.S.T.M. methods shall be mutually agreed upon by the purchaser and the seller.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: K45-1941

Standard Specifications for

TITANIUM DIOXIDE PIGMENTS[1]

A.S.T.M. Designation: D 476 – 41

ADOPTED, 1939; REVISED, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 476; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover titanium dioxide pigments, as follows:

Titanium dioxide, commercially pure.

Titanium-Barium Pigment, a pigment consisting of titanium dioxide and barium sulfate.

Titanium-Calcium Pigment, a pigment consisting of titanium dioxide and anhydrous calcium sulfate.

Titanium-Magnesium Pigment, a pigment consisting of titanium dioxide and magnesium silicates with or without added micaceous silicates.

TABLE I.—REQUIREMENTS FOR COMPOSITION.

	Titanium Dioxide Pigment	Titanium Barium Pigment		Titanium Calcium Pigment	Titanium Magnesium Pigment
		Class A	Class B		
Titanium dioxide, min., per cent	97.0	28.0	24.0	28.0	28.0
Alumina, max., per cent	....	.....	.....	.....	7.5
Matter soluble in water, max., per cent	1.5	1.5	1.5	.....	.....
Moisture and other volatile matter, max., per cent	0.5	0.5	0.5	0.5	1.0
Barium sulfate, min	....	98 per cent of remainder	98 per cent of remainder	.....	.....
Anhydrous calcium sulfate, min	....	.....	.....	98 per cent of remainder	.....
Magnesium and aluminum silicates, min.	....	.....	.....	.....	98 per cent of remainder
Coarse particles (total residue retained on a No. 325 (44-micron) sieve), max., per cent	1.0	1.0	1.0	1.0	2.0

Composition and Properties

2. (*a*) The pigments shall conform to the requirements for composition prescribed in Table I.

(*b*) In such physical properties as are specified by the purchaser, the pigment shall satisfactorily match a reference sample mutually agreed upon by the purchaser and the seller.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1938 to 1939, being revised in 1939.

These specifications are in effect a revision and consolidation of, and replace the former Standard Specifications for Titanium Barium Pigment (D 382 – 35), Titanium Calcium Pigment (D 383 – 35), Titanium Dioxide (D 384 – 36), and the Tentative Specifications for Titanium Magnesium Pigment (D 442 – 37 T) which were accordingly discontinued in 1939.

Number of Tests

3. Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10,000 lb., except that for shipments of less than 10,000 lb. two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

Methods of Testing

4. Tests shall be conducted in accordance with the appropriate methods of the American Society for Testing Materials, where applicable. Test procedures not covered by A.S.T.M. methods shall be mutually agreed upon by the purchaser and the seller.

Standard Specifications for

ZINC SULFIDE PIGMENTS[1]

A.S.T.M. Designation: D 477 - 41

ADOPTED, 1939; REVISED, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 477; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover zinc sulfide pigments, as follows:

Zinc Sulfide, commercially pure.

Zinc Sulfide - Barium Pigment, a pigment consisting of zinc sulfide and barium sulfate as indicated in Table I.

Zinc Sulfide - Magnesium Pigment, a pigment consisting of zinc sulfide and magnesium silicates with or without added micaceous silicates.

Lithopone, a pigment consisting of zinc sulfide and barium sulfate in approximately molecular ratio.

Titanated Lithopone, a pigment consisting of zinc sulfide, titanium dioxide, and barium sulfate.

TABLE I.—REQUIREMENTS FOR COMPOSITION.[3]

	Zinc Sulfide	Zinc Sulfide Barium Pigment	Zinc Sulfide Magnesium Pigment	Lithopone	Titanated Lithopone
Zinc sulfide, min., per cent	97.0	45.0	45.0	26.0	....
Zinc oxide, max., per cent	1.0	1.0	1.0	1.0	1.0
Zinc sulfide plus titanium dioxide, min., per cent	....	....	....	....	35.0
Titanium dioxide, min., per cent	....	....	....	....	13.0
Alumina, max., per cent	....	....	5.5	....	....
Matter soluble in water, max., per cent	1.5	0.8	1.0	0.8	0.8
Moisture and other volatile matter, max., per cent	0.5	0.5	1.0	0.5	0.5
Barium sulfate, min.	....	98 per cent of remainder	....	98 per cent of remainder	98 per cent of remainder
Magnesium silicates, with or without added micaceous silicates, min.	....	....	98 per cent of remainder	....	....
Coarse particles:					
Total residue retained on a No. 100 (149-micron) sieve, max., per cent	....	....	0.1	....	....
Total residue retained on a No. 325 (44-micron) sieve, max., per cent	1.0	1.0	3.0	1.0	1.0

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1938 to 1939.

These specifications comprise a consolidation and revision of the former Standard Specifications for Lithopone (D 208 - 26), Zinc Sulfide-Barium Pigment (D 285 - 36), Zinc Sulfide (D 386 - 36), and the Tentative Specifications for Zinc Sulfide Magnesium Pigment (D 443 - 37 T) which were accordingly discontinued in 1939.

[3] Editorially revised in 1944.

A "tentative revision" of this standard appears in the Tentative Revisions of Standards section at the back of this book.

Composition and Properties

2. (*a*) The pigments shall conform to the requirements for composition prescribed in Table I.

(*b*) In such physical properties as are specified by the purchaser, the pigment shall satisfactorily match a reference sample mutually agreed upon by the purchaser and the seller.

Number of Tests

3. Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10,000 lb., except that for shipments of less than 10,000 lb. two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

Methods of Testing

4. Tests shall be conducted in accordance with the appropriate methods of the American Society for Testing Materials, where applicable. Test procedures not covered by A.S.T.M. methods shall be mutually agreed upon by the purchaser and the seller.

Standard Specifications for

BARIUM SULFATE PIGMENTS[1]

A.S.T.M. Designation: D 602 – 42

ADOPTED, 1942.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 602; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover the barium sulfate pigments commercially known as barytes and blanc fixe.

Composition and Properties

2. (*a*) The pigment shall consist of barium sulfate ($BaSO_4$) without any admixture of other materials in the case of blanc fixe, and without any admixture of other materials not naturally occurring in the barite ore in the case of barytes, and which conform to the following requirements:

	Barytes	Blanc Fixe
Barium sulfate, min., per cent	94.0	97.0
Ferric oxide, max., per cent	0.05	0.02
pH, min.	3.5	3.5
Matter soluble in water, max., per cent	0.2	0.2
Moisture and other volatile matter, max., per cent	0.5	0.5
Coarse particles (total residue retained on a No. 325 (44-micron) sieve), max., per cent	0.5	0.5
Free silica (quartz, clays, or other foreign materials), max., per cent	2.0	2.0

(*b*) In such physical properties as are specified by the purchaser, the pigment shall satisfactorily match a reference sample mutually agreed upon by the purchaser and the seller.

Number of Tests

3. Two samples shall be taken at ranrom from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10,000 lb., except that for shipments of less than 10,000 lb. two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

Methods of Testing

4. Tests shall be conducted in accordance with the appropriate methods of the American Society for Testing Materials, where applicable. Test procedures not covered by A.S.T.M. methods shall be mutually agreed upon by the purchaser and the seller.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1941 to 1942.

Standard Specifications for

ALUMINUM SILICATE PIGMENT[1]

A.S.T.M. Designation: D 603 – 42

ADOPTED, 1942.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 603; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover the aluminum silicate pigment commercially known as china clay.

Composition and Properties

2. (*a*) The pigment shall be made by dry grinding and air floating, or by wet grinding a natural aluminum silicate obtained by underground mining, and shall conform to the following requirements:

Silicon dioxide, per cent	43.0 to 47.0
Aluminum oxide, per cent	37.0 to 40.0
Loss on ignition, per cent	10.0 to 15.0
Moisture and other volatile matter, max., per cent	1.0
Coarse particles (total residue retained on a No. 325 (44-micron) sieve), max., per cent	2.0

(*b*) The color shall be equal to that of a reference sample mutually agreed upon by the purchaser and the seller.

Number of Tests

3. Two samples shall be taken at ranrom from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10,000 lb., except that for shipments of less than 10,000 lb. two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

Methods of Testing

4. Tests shall be conducted in accordance with the appropriate methods of the American Society for Testing Materials, where applicable. Test procedures not covered by A.S.T.M. methods shall be mutually agreed upon by the purchaser and the seller.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1941 to 1942.

Standard Specifications for

DIATOMACEOUS SILICA PIGMENT[1]

A.S.T.M. Designation: D 604 – 42

ADOPTED, 1942.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 604; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover two types of diatomaceous silica pigments for use in paints:

Type A.—Standard fineness for general paint use.

Type B.—Extra fine, for special uses.

NOTE.—Silica pigments originating from crystalline rocks or sands are not covered by these specifications.

Composition and Properties

2. (*a*) The pigment shall be a diatomaceous silica especially manufactured and selected for use in paints by chemical, heat, and grading processes, and shall conform to the following requirements:

Loss on ignition, max., per cent	1.0
Matter soluble in HCl (1:2), max., per cent	3.0
Moisture and other volatile matter, max., per cent	1.0
Volume of settling pigment in petroleum spirits after 1 hr., min., ml.:	
Type A	35
Type B	25
Coarse particles (total residue retained on a No. 325 (44-micron) sieve), per cent:	
Type A	5.0 to 15.0
Type B	1.0 max.

(*b*) The color shall be equal to that of a reference sample mutually agreed upon by the purchaser and the seller.

Number of Tests

3. Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10,000 lb., except that for shipments of less than 10,000 lb. two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

Methods of Testing

4. Tests shall be conducted in accordance with the appropriate methods of the American Society for Testing Materials, where applicable. Test procedures not covered by A.S.T.M. methods shall be mutually agreed upon by the purchaser and the seller.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1941 to 1942.

Standard Specifications for

MAGNESIUM SILICATE PIGMENT[1]

A.S.T.M. Designation: D 605 - 42

ADOPTED, 1942.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 605; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover the magnesium silicate pigments available commercially in both fibrous and granular form.

Composition and Properties

2. (*a*) The pigment shall be made by grinding natural magnesium silicate obtained by underground mining, and shall conform to the following requirements:

Silicon dioxide, per cent	40.0 to 56.0
Magnesium oxide, per cent	20.0 to 32.0
Loss on ignition, per cent	4.0 to 7.0
Moisture and other volatile matter, max., per cent	1.0
Coarse particles (total residue retained on a No. 325 (44-micron) sieve), max., per cent	2.0

(*b*) The color shall be equal to that of a reference sample mutually agreed upon by the purchaser and the seller.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1941 to 1942, being revised in 1942.

Number of Tests

3. Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10,000 lb., except that for shipments of less than 10,000 lb. two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

Methods of Testing

4. Tests shall be conducted in accordance with the appropriate methods of the American Society for Testing Materials, where applicable. Test procedures not covered by A.S.T.M. methods shall be mutually agreed upon by the purchaser and the seller.

Standard Specifications for

LEAD TITANATE[1]

A.S.T.M. Designation: D 606 – 42

ADOPTED, 1942.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 606; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover the pigment commercially known as lead titanate.

Composition and Properties

2. (*a*) The pigment shall consist of lead titanate with a minor amount of lead sulfate, and shall conform to the following requirements:

Lead titanate ($PbTiO_3$), min., per cent	88.0
Lead sulfate ($PbSO_4$), max., per cent	9.0
Lead monoxide (PbO), uncombined, max., per cent	1.0
Total titanium calculated as TiO_2, min., per cent	23.0
Titanium dioxide (TiO_2), uncombined, max., per cent	3.0
Matter soluble in water, max., per cent	1.5
Moisture and other volatile matter, max., per cent	0.5
Coarse particles (total residue retained on a No. 325 (44-micron) sieve), max., per cent	1.0

(*b*) In such physical properties as are specified by the purchaser, the pigment shall satisfactorily match a reference sample mutually agreed upon by the purchaser and the seller.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1941 to 1942.

Number of Tests

3. Two samples shall be taken at ranrom from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10,000 lb., except that for shipments of less than 10,000 lb. two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

Methods of Testing

4. Tests shall be conducted in accordance with the appropriate methods of the American Society for Testing Materials, where applicable. Test procedures not covered by A.S.T.M. methods shall be mutually agreed upon by the purchaser and the seller.

Standard Specifications for

MICA PIGMENT[1]

A.S.T.M. Designation: D 607 – 42

ADOPTED, 1942.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 607; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover the finely divided mica commercially known as pigment mica, suitable for use in the manufacture of protective coatings.

Composition and Properties

2. (*a*) The pigment shall be made by wet grinding muscovite mica, and shall conform to the following requirements:

Apparent density, max., lb. per cu. ft.	10.0
Moisture and other volatile matter, max., per cent	0.5
Grit, max., per cent	0.15
Coarse particles, max., per cent:	
Total residue retained on a No. 140 (105-micron) sieve	0.1
Total residue retained on a No. 325 (44-micron) sieve	7.0

(*b*) The color shall be equal to that of a reference sample mutually agreed upon by the purchaser and the seller.

Number of Tests

3. Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10,000 lb., except that for shipments of less than 10,000 lb. two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

Methods of Testing

4. Tests shall be conducted in accordance with the appropriate methods of the American Society for Testing Materials, where applicable. Test procedures not covered by A.S.T.M. methods shall be mutually agreed upon by the purchaser and the seller.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1941 to 1942.

Emergency Alternate Provisions EA – D 607 applying to this standard appear in the pink section of this book.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: K26-1941

Standard Specifications for

LAMPBLACK[1]

A.S.T.M. Designation: D 209 – 41

ADOPTED, 1926; REVISED, 1930, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 209; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover the pigment commercially known as lampblack. The pigment may be purchased in the dry form or as a paste in oil or in japan.

Composition and Properties

2. (*a*) *Dry Pigment.*—The pigment shall be made by burning oils or tars in such a manner as to form a deposit of carbon or soot. It shall be high grade in every respect and shall be free from oil, greasy matter, and admixture of any other substance. The pigment shall conform to the following requirements:

Ash, max., per cent	1.0
Acetone extract, max., per cent	2.0
Coarse particles, (total residue retained on a No. 325 (44-micron) sieve), max., per cent	1.0
Tone when diluted with zinc oxide	clear-blue-gray

(*b*) *Paste in Oil.*—The paste in oil shall be made by thoroughly grinding the specified pigment with linseed oil. As received it shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions, without curdling, with linseed oil, turpentine, or volatile petroleum spirits, or any mixture of these substances. The paste shall conform to the following requirements:

Pigment, min., per cent	25
Linseed oil, max., per cent	75
Moisture and other volatile matter, max., per cent	0.7
Coarse particles and skins (total residue retained on a No. 325 (44-micron) sieve), max., per cent of the dry pigment	1.0

(*c*) *Paste in Japan.*—The paste in japan shall be made by thoroughly grinding the specified pigment in high-grade grinding japan. As received it shall not be caked in the container and shall break up readily in turpentine to form a smooth paint of brushing consistency that will dry within 1 hr. to a hard, flat coat. The paste shall conform to the following requirements:

Pigment, min., per cent	25
Vehicle (japan), max., per cent	75
Nonvolatile matter in the vehicle, min., per cent of the vehicle	40
Coarse particles and skins (total residue retained on a No. 325 (44-micron) sieve), max. per cent of the dry pigment	1.0

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1925 to 1926, being revised in 1926.

(*d*) The color and tone shall be equal to, and the tinting strength not less than, that of a reference sample mutually agreed upon by the purchaser and the seller.

Number of Tests

3. Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10,000 lb., except that for shipments of less than 10,000 lb. two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

Methods of Testing

4. Tests shall be conducted in accordance with the appropriate methods of the American Society for Testing Materials, where applicable. Test procedures not covered by A.S.T.M. methods shall be mutually agreed upon by the purchaser and the seller.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: K36-1941

Standard Specifications for

BONE BLACK[1]

A.S.T.M. Designation: D 210 – 41

ADOPTED, 1926; REVISED, 1930, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 210; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover the pigment commercially known as bone black, ivory black, or drop black. The pigment may be purchased in the dry form or as a paste in oil or in japan.

Composition and Properties

2. (*a*) *Dry Pigment.*—The pigment shall be made by the calcination of bones, shall be unmixed with any other substance, and shall conform to the following requirements:

Ash, max., per cent of pigment dried at 105 C	88.0
Ash insoluble in acids, max., per cent	1.0
Acetone extract, max., per cent	2.0
Coarse particles (total residue retained on a No. 325 (44-micron) sieve), max., per cent	2.0
Tone when diluted with zinc oxide	clear-blue-gray

(*b*) *Paste in Oil.*—The paste in oil shall be made by thoroughly grinding the specified pigment with linseed oil. As received it shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions, without curdling, with linseed oil, turpentine, or volatile petroleum spirits, or any mixture of these substances. The paste shall conform to the following requirements:

Pigment, min., per cent	45
Linseed oil, max., per cent	55
Coarse particles and skins (total residue retained on a No. 325 (44-micron) sieve), max., per cent of the dry pigment	2.5

(*c*) *Paste in Japan.*—The paste in japan shall be made by thoroughly grinding the pigment in high-grade grinding japan. As received it shall not be caked in the container and shall break up readily in turpentine to form a smooth paint of brushing consistency that will dry within 1 hr. to a hard, flat coat. The paste shall conform to the following requirements:

Pigment, min., per cent	45
Vehicle (japan), max., per cent	55
Nonvolatile matter in the vehicle, min., per cent of the vehicle	40
Coarse particles and skins (total residue retained on a No. 325 (44-micron) sieve), max., per cent of the dry pigment	2.5

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1925 to 1926, being revised in 1926.

(*d*) The color and tone shall be equal to, and the tinting strength not less than, that of a reference sample mutually agreed upon by the purchaser and the seller.

Number of Tests

3. Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10,000 lb., except that for shipments of less than 10,000 lb. two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

Methods of Testing

4. Tests shall be conducted in accordance with the appropriate methods of the American Society for Testing Materials, where applicable. Test procedures not covered by A.S.T.M. methods shall be mutually agreed upon by the purchaser and the seller.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: K48-1941

Standard Specifications for

BLUE LEAD; BASIC SULFATE[1]

A.S.T.M. Designation: D 405 - 41

ADOPTED, 1938; REVISED, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 405; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover the pigment commercially known as blue lead, basic sulfate, suitable for use in paints. The pigment may be purchased in the dry form or as a paste in oil.

Composition and Properties

2. (*a*) *Dry Pigment.*—The dry pigment shall consist of basic lead sulfate and certain other ingredients, which upon analysis shall conform to the following requirements:

Lead sulfate, min., per cent	45.0
Lead oxide, min., per cent	30.0
Lead sulfide, max., per cent	12.0
Lead sulfite, max., per cent	5.0
Zinc oxide, max., per cent	5.0
Carbon and undetermined matter, max., per cent	5.0
Coarse particles (total residue retained on a No. 325 (44-micron) sieve), max., per cent	1.0

(*b*) *Paste in Oil.*—The paste shall be made by thoroughly grinding the pigment with linseed oil. The paste as received shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions, without curdling, with linseed oil, turpentine, or volatile petroleum spirits or any mixture of these substances. The paste shall conform to the following requirements:

Pigment, min., per cent	89.0
Linseed oil, max., per cent	11.0
Moisture and other volatile matter, max., per cent	0.7
Coarse particles and skins (total residue retained on a No. 325 (44-micron) sieve), max., per cent of the dry pigment	1.5

Number of Tests

3. Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10,000 lb., except that for shipments of less than 10,000 lb. two samples shall be taken. At the option of the purchaser, the

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1935 to 1938, being revised in 1938.

samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

Methods of Testing

4. Tests shall be conducted in accordance with the appropriate methods of the American Society for Testing Materials, where applicable. Test procedures not covered by A.S.T.M. methods shall be mutually agreed upon by the purchaser and the seller.

Standard Specifications for

CARBON BLACK[1]

A.S.T.M. Designation: D 561 – 41

ADOPTED, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 561; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover the pigment commercially known as carbon black, suitable for use in the manufacture of protective or decorative coatings.

Composition and Properties

2. (*a*) The pigment shall be made by burning natural gas in such a manner as to form a deposit of carbon, but without any admixtures, and shall conform to the following requirements:

Ash, max., per cent	0.2
Acetone extract, max., per cent	0.5
True grit (total residue retained on a No. 325 (44-micron) sieve), max., per cent	0.2
Moisture and other volatile matter, max., per cent	8.0[a]

[a] It may be necessary for the purchaser and the seller to agree upon a higher maximum moisture content in high-color blacks.

(*b*) The color shall be equal to, and the tinting strength (Note) not less than, that of a reference sample mutually agreed upon by the purchaser and the seller.

NOTE.—For the tinting strength test a ratio of 100 parts of white to 1 part of black is usually suitable.

Number of Tests

3. Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10,000 lb., except that for shipments of less than 10,000 lb. two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

NOTE.—High-color blacks are very hygroscopic and should be protected against atmospheric moisture during storage.

Methods of Testing

4. Tests shall be conducted in accordance with the appropriate methods of the American Society for Testing Materials, where applicable. Test procedures not covered by A.S.T.M. methods shall be mutually agreed upon by the purchaser and the seller.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1940 to 1941, being revised in 1941.

Standard Specifications for

OCHER[1]

A.S.T.M. Designation: D 85 - 41

ADOPTED, 1924; REVISED, 1927, 1940, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 85; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover ferrous earthy pigments included under the general term "ocher". The pigment may be purchased in the dry form or as a paste in oil or in japan.

Composition and Properties

2. (*a*) *Dry Pigment.*—The pigment shall be a hydrated oxide of iron permeating a siliceous base, and shall be free from added impurities and added coloring matter. The pigment shall conform to the following requirements:

Ferric oxide, min., per cent	17
Calcium oxide, max., per cent	5
Lead chromate	none
Organic coloring matter	none
Moisture and other volatile matter, max., per cent	1.0
Coarse particles (total residue retained on a No. 325 (44-micron) sieve), max., per cent	1.0

(*b*) *Paste in Oil.*—The paste in oil shall be made by thoroughly grinding the specified pigment with linseed oil. As received it shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions, without curdling, with linseed oil, turpentine, or volatile petroleum spirits, or any mixture of these substances. The paste shall conform to the following requirements:

Pigment, per cent	69 to 73
Linseed oil, per cent	27 to 31
Moisture and other volatile matter, max., per cent	0.7
Coarse particles and skins (total residue retained on a No. 325 (44-micron) sieve), max., per cent of the dry pigment	1.5

(*c*) *Paste in Japan.*—The paste in japan shall be made by thoroughly grinding the specified pigment in high-grade grinding japan. As received it shall not be caked in the container and shall break up readily in turpentine to form a smooth paint of brushing consistency that will dry within 1 hr. to a hard, flat coat that can be varnished within 5 hr. of the time of application

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1921 to 1924, being revised in 1924.

without streaking or bleeding. The paste shall conform to the following requirements:

Pigment, per cent	69 to 73
Vehicle (japan), per cent	27 to 31
Nonvolatile matter in vehicle, min., per cent of the vehicle	40
Coarse particles and skins (total residue retained on a No. 325 (44-micron) sieve), max., per cent of the dry pigment	1.5

(*d*) The mass color and character of the tint formed by mixture with a white pigment shall be the same as, and the strength not less than, that of a reference sample mutually agreed upon by the purchaser and the seller.

Number of Tests

3. Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10,000 lb., except that for shipments of less than 10,000 lb. two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

Methods of Testing

4. Tests shall be conducted in accordance with the appropriate methods of the American Society for Testing Materials, where applicable. Test procedures not covered by A.S.T.M. methods shall be mutually agreed upon by the purchaser and the seller.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO: K29-1941

AMERICAN ASSOCIATION STATE
HIGHWAY OFFICIALS STANDARD
A.A.S.H.O. NO.: M 131-42

Standard Specifications for
PRUSSIAN BLUE[1]

A.S.T.M. Designation: D 261 – 41

ADOPTED, 1928; REVISED, 1939, 1940, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 261; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover the pigment commercially known as Prussian blue, Chinese blue, or iron blue. The pigment may be purchased in the dry form or as a paste in oil or in japan.

Composition and Properties

2. (*a*) *Dry Pigment.*—The pigment shall be the blue product formed by the reaction of solutions of iron salts with ferrocyanide or ferricyanide solution. It shall not be admixed with any other substance. The pigment shall conform to the following requirements:

Total matter soluble in water, max., per cent	0.5
Moisture and other volatile matter, max., per cent	7.0

(*b*) *Paste in Oil.*—The paste in oil shall be made by thoroughly grinding the specified pigment with linseed oil. The paste as received shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions, without curdling, with linseed oil, turpentine, or volatile petroleum spirits, or any mixture of these substances. The paste shall conform to the following requirements:

Pigment, min., per cent	48.0
Linseed oil, max., per cent	52.0
Moisture and other volatile matter, max., per cent	3.5
Coarse particles and skins (total residue retained on a No. 325 (44-micron) sieve), max., per cent of the dry pigment	1.5

(*c*) *Paste in Japan.*—The paste in japan shall be made by thoroughly grinding the specified pigment in high-grade grinding japan. As received it shall not be caked in the container and shall break up readily in turpentine to form a smooth paint of brushing consistency that will dry within 1 hr. to a hard flat coat that can be varnished within 5 hr. of the time of application without streaking or bleeding. The paste shall conform to the following requirements:

Pigment, min., per cent	48.0
Vehicle (japan), max., per cent	52.0
Nonvolatile matter in the vehicle, min., per cent of the vehicle	40.0
Coarse particles and skins (total residue retained on a No. 325 (44-micron) sieve), max., per cent of the dry pigment	1.5

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1927 to 1928, being revised in 1928.

(*d*) The mass color and character of the tint formed by mixture with a white pigment shall be the same as, and the strength not less than, that of a reference sample mutually agreed upon by the purchaser and the seller.

NOTE.—The physical properties and tests of Prussian blue, particularly the tinting strength, are considered a better measure of value than the percentages of chemical constituents.

Number of Tests

3. Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10,000 lb., except that for shipments of less than 10,000 lb. two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

Methods of Testing

4. Tests shall be conducted in accordance with the appropriate methods of the American Society for Testing Materials, where applicable. Test procedures not covered by A.S.T.M. methods shall be mutually agreed upon by the purchaser and the seller.

Standard Specifications for

ULTRAMARINE BLUE[1]

A.S.T.M. Designation: D 262 – 41

ADOPTED, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 262; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover the pigments commercially known as ultramarine blue, suitable for use in paints, enamels, lacquers, and similar products. The pigment may be purchased in the dry form or as a paste in oil or in japan.

Composition and Properties

2. (*a*) *Dry Pigment.*—The pigments shall be manufactured ultramarine blue obtained by the furnacing of mixtures of clays and silicas with sodium salts, sulfur, and carbonaceous material, and having a chemical composition[3] which will generally fall within the following range, expressing the elements except sulfur as their oxides:

Sodium oxide, per cent	18 to 23
Aluminum oxide, per cent	20 to 32
Silicon dioxide, per cent	35 to 50
Sulfur, per cent	8 to 14

The pigment shall conform to the following requirements (Note):

Moisture and other volatile matter, max., per cent	4.0
Coarse particles (total residue retained on a No. 325 (44-micron) sieve), max., per cent	1.0

(*b*) *Paste in Oil.*—The paste in oil shall be made by thoroughly grinding the specified pigment with linseed oil. As received it shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions, without curdling, with linseed oil, turpentine, or volatile petroleum spirits, or any mixture of these substances. The paste shall conform to the following requirements (Note):

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to their present adoption as standard, these specifications were published as tentative from 1927 to 1928, being revised in 1928. They were adopted in 1928, published as standard from 1928 to 1940, but withdrawn and republished as tentative from 1940 to 1941, being revised in 1941.

[3] The current view is that the ultramarine blues all possess substantially the same type of molecular lattice or framework of definite configuration, built up of alternating atoms of oxygen and silicon or aluminum. The ratio of silicon to aluminum atoms in the lattice is variable, and to a considerable degree silicon may replace aluminum. Within the lattice are located atoms of sodium and sulfur whose number per lattice unit is also variable. The variations possible, as noted above, permit the existence of many separate members of the ultramarine blue group. All are characterized, however, by giving substantially identical patterns when examined by the use of x-rays. This pattern is described in full in *Zeitschrift fur Anorganische und Allgemeine Chemie*, Vol. 228 (1936), p. 305, and is characteristic of the ultramarine blues as a group.

Pigment, min., per cent	70.0
Linseed oil, max., per cent	30.0
Moisture and other volatile matter, max., per cent	3.0
Coarse particles and skins (total residue retained on a No. 325 (44-micron) sieve), max., per cent of the dry pigment	1.5

(*c*) *Paste in Japan.*—The paste in japan shall be made by thoroughly grinding the specified pigment in high-grade grinding japan. As received it shall not be caked in the container and shall break up readily in turpentine to form a smooth paint of brushing consistency, that will dry within 1 hr. to a hard flat coat that can be varnished within 5 hr. of the time of application without streaking or bleeding. The paste shall conform to the following requirements (Note):

Pigment, min., per cent	70.0
Vehicle (japan), max., per cent	30.0
Nonvolatile matter in the vehicle, min., per cent of the vehicle	40.0
Coarse particles and skins (total residue retained on a No. 325 (44-micron) sieve), max., per cent of the dry pigment	1.5

(*d*) The mass color and character of the tint formed by mixture with a white pigment shall be the same as, and the strength not less than, that of a reference sample mutually agreed upon by the purchaser and the seller.

NOTE.—The chemical composition has not been found to be a dependable measure of value in determining the properties of ultramarine blues. The physical properties and tests such as shade and tinting strength are the best measure of value.

Number of Tests

3. Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10,000 lb., except that for shipments of less than 10,000 lb. two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

Methods of Testing

4. Tests shall be conducted in accordance with the appropriate methods of the American Society for Testing Materials, where applicable. Test procedures not covered by A.S.T.M. methods shall be mutually agreed upon by the purchaser and the seller.

AMERICAN ASSOCIATION STATE
HIGHWAY OFFICIALS STANDARD
A.A.S.H.O. NO.: M 130-42

Standard Specifications for
PURE CHROME GREEN[1]

A.S.T.M. Designation: D 212 – 41

ADOPTED, 1927; REVISED, 1940, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 212; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover the pigment commercially known as pure chrome green. The pigment may be purchased in the dry form or as a paste in oil or in japan.

Composition and Properties

2. (*a*) *Dry Pigment.*—The pigment shall be a precipitated mixture of lead chromate and iron ferrocyanide and ferricyanide blue, with or without other insoluble compounds of lead and shall conform to the following requirements:

Of the total lead[a] in the pigment, percentage present as chromate, ($PbCrO_4$),[a] min.	70
Moisture and other volatile matter, max., per cent.	4.0
Total other impurities, max., per cent.	3.0
Coarse particles (total residue retained on a No. 325 (44-micron) sieve), max., per cent.	1.0

[a] Calculated as metallic lead.

(*b*) *Paste in Oil.*—The paste in oil shall be made by thoroughly grinding the specified pigment with linseed oil. The paste as received shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions, without curdling, with linseed oil, turpentine, or volatile petroleum spirits, or any mixture of these substances. The paste shall conform to the following requirements:

Pigment, min., per cent.	70
Linseed oil, max., per cent.	30
Moisture and other volatile matter, max., per cent.	3.0
Coarse particles and skins (total residue retained on a No. 325 (44-micron) sieve), max., per cent of the dry pigment.	1.5

(*c*) *Paste in Japan.*—The paste in japan shall be made by thoroughly grinding the specified pigment with high-grade grinding japan. The paste as received shall not be caked in the container and shall break up readily in turpentine to form a smooth paint of brushing consistency that will dry within 1 hr. to a hard, flat coat that can be varnished within 5 hr. of the time of application without streaking or bleed-

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1925 to 1927, being revised in 1926 and 1927.

ing. The paste shall conform to the following requirements:

Pigment, min., per cent	65
Vehicle (japan), max., per cent	35
Nonvolatile matter in vehicle, min., per cent of the vehicle	40
Coarse particles and skins (total residue retained on a No. 325 (44-micron) sieve), max., per cent of the dry pigment	1.5

(*d*) The mass color and character of the tint formed by mixture with a white pigment shall be the same as, and the strength not less than, that of a reference sample mutually agreed upon by the purchaser and the seller.

Number of Tests

3. Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10,000 lb., except that for shipments of less than 10,000 lb. two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

Methods of Testing

4. Tests shall be conducted in accordance with the appropriate methods of the American Society for Testing Materials, where applicable. Test procedures not covered by A.S.T.M. methods shall be mutually agreed upon by the purchaser and the seller.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: K28-1941

Standard Specifications for
REDUCED CHROME GREEN[1]

A.S.T.M. Designation: D 213 – 41

ADOPTED, 1927; REVISED, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 213; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover the pigment commercially known as reduced chrome green, also known as grinders green. The pigment may be purchased in the dry form or as a paste in oil or in japan.

Composition and Properties

2. (*a*) *Dry Pigment.*—The pigment shall be a mixture of lead chromate and iron ferrocyanide and ferricyanide blue, with or without other insoluble compounds of lead, precipitated on a base of barium sulfate or insoluble siliceous material or any mixture thereof. The pigment shall conform to the following requirements:

Sum of the barium sulfate and insoluble siliceous material, max., per cent	80
Color (total of insoluble lead compounds and iron blue), min., per cent	20
Of the total lead[a] in the pigment, percentage present as chromate ($PbCrO_4$),[a] min.	70
Total calcium oxide in any form soluble in acid, max., per cent	1.0
Moisture and other volatile matter, max., per cent	1.0
Coarse particles (total residue retained on a No. 325 (44-micron) sieve), max., per cent	1.5

[a] Calculated as metallic lead.

(*b*) *Paste in Oil.*—The paste in oil shall be made by thoroughly grinding the specified pigment with linseed oil. The paste as received shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions, without curdling, with linseed oil, turpentine, or volatile petroleum spirits, or any mixture of these substances. The paste shall conform to the following requirements:

Pigment, min., per cent	80
Linseed oil, max., per cent	20
Moisture and other volatile matter, max., per cent	1.0
Coarse particles and skins (total residue retained on a No. 325 (44-micron) sieve), max., per cent of the dry pigment	2.0

(*c*) *Paste in Japan.*—The paste in japan shall be made by thoroughly grinding the specified pigment in high-grade grinding japan. The paste as

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1925 to 1927, being revised in 1926 and 1927.

received shall not be caked in the container and shall break up readily in turpentine to form a smooth paint of brushing consistency that will dry within 1 hr. to a hard flat coat that can be varnished within 5 hr. of the time of application without streaking or bleeding. The paste shall conform to the following requirements:

Pigment, min., per cent	75
Vehicle (japan), max., per cent	25
Nonvolatile matter in vehicle, min, per cent of the vehicle	40
Coarse particles and skins (total residue retained on a No. 325 (44-micron) sieve), max., per cent of the dry pigment.	2.0

(*d*) The mass color and character of the tint formed by mixture with a white pigment shall be the same as, and the strength not less than, that of a reference sample mutually agreed upon by the purchaser and the seller.

Number of Tests

3. Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10,000 lb., except that for shipments of less than 10,000 lb. two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

Methods of Testing

4. Tests shall be conducted in accordance with the appropriate methods of the American Society for Testing Materials, where applicable. Test procedures not covered by A.S.T.M. methods shall be mutually agreed upon by the purchaser and the seller.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: K37-1941

Standard Specifications for
CHROME OXIDE GREEN[1]

A.S.T.M. Designation: D 263 – 41

ADOPTED, 1928; REVISED, 1940, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 263; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover the pigment commercially known as chrome oxide green. The pigment may be purchased in the dry form or as a paste in oil.

Composition and Properties

2. (*a*) *Dry Pigment.*—The pigment shall consist of practically pure chromic oxide (Cr_2O_3) without any admixture, and shall conform to the following requirements:

Total chromium (calculated as Cr_2O_3), min., per cent	97.0
Moisture and other volatile matter, max., per cent	0.5
Coarse particles (total residue retained on a No. 325 (44-micron) sieve), max., per cent	2.0

(*b*) *Paste in Oil.*—The paste shall be made by thoroughly grinding the specified pigment with linseed oil. As received it shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions, without curdling, with linseed oil, turpentine, or volatile petroleum spirits, or any mixture of these substances. The paste shall conform to the following requirements:

Pigment, min., per cent	75
Linseed oil, max., per cent	25
Moisture and other volatile matter, max., per cent	0.5
Coarse particles and skins (total residue retained on a No. 325 (44-micron) sieve), max., per cent of the dry pigment	2.5

(*c*) The mass color and character of the tint formed by mixture with a white pigment shall be the same as, and the strength not less than, that of a reference sample mutually agreed upon by the purchaser and the seller.

Number of Tests

3. Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1927 to 1928, being revised in 1928.

ratio of two samples for each 10,000 lb., except that for shipments of less than 10,000 lb. two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

Methods of Testing

4. Tests shall be conducted in accordance with the appropriate methods of the American Society for Testing Materials, where applicable. Test procedures not covered by A.S.T.M. methods shall be mutually agreed upon by the purchaser and the seller.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: K27.1-1943

Standard Specifications for

CHROME YELLOW AND CHROME ORANGE[1]

A.S.T.M. Designation: D 211-43

ADOPTED, 1943.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 211; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover five types of commercially pure lead chromate pigments, as follows:

Type I.—Primrose chrome yellow.
Type II.—Lemon chrome yellow.
Type III.—Medium chrome yellow.
Type IV.—Light chrome orange.
Type V.—Dark chrome orange.

The pigment may be purchased in the dry form or as a paste in oil or in japan.

Composition and Properties

2. (*a*) *Dry Pigments.*—The pigments shall be chemical precipitates consisting of normal or basic lead chromates or mixtures of these with or without admixtures of other insoluble compounds of lead, but without other admixtures except those of reagent materials introduced specifically to improve those properties for which the pigment is used. The pigments shall be free of extenders (barium sulfate, clay, magnesium silicate, whiting, etc.) and shall conform to the requirements for composition prescribed in Table I.

TABLE I.—REQUIREMENTS FOR COMPOSITION.

	Type I	Type II	Type III	Type IV	Type V
Lead chromate ($PbCrO_4$), min., per cent	50.0	68.0	93.0	60.0	55.0
Total matter soluble in water, max., per cent	1.0	1.0	1.0	1.0	1.0
Total of all substances other than insoluble compounds of lead, max., per cent	8.0	8.0	7.0	3.0	3.0
Moisture and other volatile matter, max., per cent	1.0	1.0	1.0	1.0	1.0
Coarse particles (total residue retained on a No. 325 (44-micron) sieve), max., per cent	1.0	1.0	1.0	1.0	1.0
Organic colors and lakes	none	none	none	none	none

(*b*) *Paste in Oil.*—The paste in oil shall be made by thoroughly grinding the specified pigment with linseed oil. As received it shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions, without curdling, with linseed oil, turpentine, or volatile petroleum spirits, or any mixture of these sub-

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to their present adoption as standard, these specifications were published as tentative from 1925 to 1927, being revised in 1926 and 1927. They were adopted in 1927, published as standard from 1927 to 1942, being revised in 1940 and 1941; but withdrawn, revised, and republished as tentative from 1942 to 1943.

stances. The paste shall conform to the following requirements:

Pigment, min., per cent	75.0
Linseed oil, max., per cent	25.0
Moisture and other volatile matter, max., per cent	1.0
Coarse particles and skins (total residue retained on a No. 325 (44-micron) sieve), max., per cent of the dry pigment	1.5

(*c*) *Paste in Japan.*—The paste in japan shall be made by thoroughly grinding the specified pigment with high-grade grinding japan. As received it shall not be caked in the container and shall break up readily in turpentine to form a smooth paint of brushing consistency that will dry within 1 hr. to a hard, flat coat that can be varnished within 5 hr. of the time of application without streaking or bleeding. The paste shall conform to the following requirements:

Pigment, min., per cent	70.0
Vehicle (japan), max., per cent	30.0
Nonvolatile matter in the vehicle, min., per cent of the vehicle	40.0
Coarse particles and skins (total residue retained on a No. 325 (44-micron) sieve), max., per cent of the dry pigment	1.5

(*d*) The mass color and character of the tint formed by mixture with a white pigment shall be the same as, and the strength not less than, that of a reference sample mutually agreed upon by the purchaser and the seller.

Number of Tests

3. Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10,000 lb., except that for shipments of less than 10,000 lb. two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

Methods of Testing

4. Tests shall be conducted in accordance with the appropriate methods of the American Society for Testing Materials, where applicable. Test procedures not covered by A.S.T.M. methods shall be mutually agreed upon by the purchaser and the seller.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: K50-1941

Standard Specifications for

C.P. ZINC YELLOW (ZINC CHROMATE)[1]

A.S.T.M. Designation: D 478 - 41

ADOPTED, 1939; REVISED, 1940, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 478; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover the pigment commercially known as chemically pure zinc yellow or zinc chromate. The pigment may be purchased in the dry form or as a paste in oil.

Composition and Properties

2. (*a*) *Dry Pigment.*—The pigment shall consist of a complex salt of zinc and potassium chromates. It shall be free from any lead compound, any organic coloring matter, or any substrate of any kind, and shall be completely soluble in acetic acid (10 per cent) at a temperature of 80 C. The pigment shall conform to the following requirements:

Zinc oxide, max., per cent	40.0
Chromic anhydride, CrO_3, min., per cent	41.0
Alkaline salts, calculated as K_2O, per cent	9.0 to 13.0
Combined water, max., per cent	8.0
Moisture and other volatile matter, max., per cent	1.0
Coarse particles (total residue retained on a No. 325 (44-micron) sieve), max., per cent	1.0

(*b*) *Paste in Oil.*—The paste shall be a dispersion of the specified pigment in linseed oil. As received it shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions, without curdling, with linseed oil, turpentine, or volatile petroleum spirits, or any mixture of these substances. The paste shall conform to the following requirements:

Pigment, min., per cent	74.0
Linseed oil, max., per cent	26.0
Moisture and other volatile matter, max., per cent	0.8
Coarse particles and skins (total residue retained on a No. 325 (44-micron) sieve), max., per cent of the dry pigment	1.5

(*c*) The mass color and character of the tint formed by mixture with a white pigment the same as, and the strength not less than, that of a reference sample mutually agreed upon by the purchaser and the seller.

Number of Tests

3. Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1938 to 1939.

unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10,000 lb., except that for shipments of less than 10,000 lb. two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

Methods of Testing

4. Tests shall be conducted in accordance with the appropriate methods of the American Society for Testing Materials, where applicable. Test procedures not covered by A.S.T.M. methods shall be mutually agreed upon by the purchaser and the seller.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: K24-1941

Standard Specifications for

RED LEAD[1]

A.S.T.M. Designation: D 83 - 41

ADOPTED, 1924; REVISED, 1931, 1939, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 83; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover three grades of red pigment commercially known as red lead. The pigment may be purchased in the dry form or as a paste in oil.

Composition and Properties

2. (*a*) *Dry Pigment.*—The pigment shall be made by roasting litharge or metallic lead, or compounds of lead that yield litharge by heating, and shall consist entirely of oxides of lead, free from adulterants. The three grades of pigment shall conform to the following requirements:

True red lead (Pb_3O_4), min., per cent:	
85 per cent grade	85.0
95 per cent grade	95.0
97 per cent grade	97.0
Total impurities including moisture, water soluble matter, and matter insoluble in a mixture of nitric acid and hydrogen peroxide, max., per cent	1.0
Lead monoxide, PbO	remainder
Coarse particles (total residue retained on a No. 325 (44-micron) sieve), max., per cent	1.0

When mixed as indicated in the following table, the resulting paint, brushed on a smooth vertical iron surface, shall dry hard and elastic without running, streaking, or sagging:

Dry red lead	20 lb.
Raw linseed oil	5 pt.
Turpentine	2 gills
Liquid drier	2 gills

(*b*) *Paste in Oil.*—The paste shall be made by thoroughly grinding the specified pigment with linseed oil (Note). The paste as shipped by the seller, and for three months thereafter, shall not be caked in the container, and shall break up readily in oil to form a smooth paint of brushing consistency. The paste shall conform to the following requirements:

Pigment, per cent	92.0 to 94.0
Linseed oil, per cent	6.0 to 8.0
Moisture and other volatile matter, max., per cent	0.5
Coarse particles and skins (total residue retained on a No. 325 (44-micron) sieve), max., per cent of the dry pigment	1.5

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1921 to 1924, being revised in 1924.

When mixed as indicated in the following table, the resulting paint, brushed on a smooth, vertical iron surface, shall dry hard and elastic without running, streaking, or sagging:

Red lead paste	20 lb.
Raw linseed oil	3 pt.
Turpentine	2 gills
Liquid drier	2 gills

NOTE.—Purchasers are cautioned that 85 per cent grade red lead should not be bought in paste form. The 95 per cent grade, if made into paste, should be used within a short period of time after grinding. When pure red lead paste is to be stored for a considerable period of time, the 97 per cent grade of red lead should be specified. The storage of paste red lead in places of high temperature should be avoided, as heat accelerates the tendency of this material to cake or harden.

Number of Tests

3. Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10,000 lb., except that for shipments of less than 10,000 lb. two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

Methods of Testing

4. Tests shall be conducted in accordance with the appropriate methods of the American Society for Testing Materials, where applicable. Test procedures not covered by A.S.T.M. methods shall be mutually agreed upon by the purchaser and the seller.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: K31-1941

Standard Specifications for

REDUCED PARA RED[1]

A.S.T.M. Designation: D 264 – 41

ADOPTED, 1928; REVISED, 1939, 1940, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 264; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover the red pigment commercially known as reduced para red. The pigment may be purchased in the dry form or as a paste in oil or in japan.

Composition and Properties

2. (*a*) *Dry Pigment.*—The pigment shall consist of a mixture of para-nitraniline toner and substrate, made either by precipitating the toner on the substrate or by mixing slurries. Unless otherwise specified by the purchaser, the substrate shall be calcium carbonate. The pigment shall conform to the following requirements:

Pure organic coloring matter (para red), min., per cent	15.0
Calcium carbonate, max., per cent	85.0
Moisture and other volatile matter, max., per cent	1.0
Coarse particles (total residue retained on a No. 325 (44-micron) sieve), max., per cent	1.5

(*b*) *Paste in Oil.*—The paste in oil shall be a dispersion of the specified pigment in linseed oil. As received it shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions, without curdling, with linseed oil, turpentine, or volatile petroleum spirits, or any mixture of these substances. The paste shall conform to the following requirements:

Pigment, min., per cent	74.0
Linseed oil, max., per cent	26.0
Moisture and other volatile matter, max., per cent	0.7
Coarse particles and skins (total residue retained on a No. 325 (44-micron) sieve), max., per cent of the dry pigment	2.0

(*c*) *Paste in Japan.*—The paste in japan shall be a dispersion of the specified pigment in high-grade grinding japan. As received it shall not be caked in the container and shall break up readily in turpentine to form a smooth paint of brushing consistency that will dry within 1 hr. to a hard flat coat that can be varnished within 5 hr. after the time of application. The paste

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1927 to 1928, being revised in 1928.

shall conform to the following requirements:

Pigment, min., per cent	74.0
Vehicle (japan), max., per cent	26.0
Nonvolatile matter in the vehicle, min., per cent of the vehicle	40.0
Coarse particles and skins (total residue retained on a No. 325 (44-micron) sieve), max., per cent of the dry pigment	2.0

(*d*) The mass color and character of the tint formed by mixture with a white pigment shall be the same as, and the strength not less than, that of a reference sample mutually agreed upon by the purchaser and the seller.

Number of Tests

3. Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10,000 lb., except that for shipments of less than 10,000 lb. two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

Methods of Testing

4. Tests shall be conducted in accordance with the appropriate methods of the American Society for Testing Materials, where applicable. Test procedures not covered by A.S.T.M. methods shall be mutually agreed upon by the purchaser and the seller.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: K49-1941

Standard Specifications for

C.P. PARA RED TONER[1]

A.S.T.M. Designation: D 475 - 41

ADOPTED, 1939; REVISED, 1940, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 475; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover the red pigment commercially known as "chemically pure" para red toner. The pigment may be purchased in the dry form or as a paste in oil.

Composition and Properties

2. (*a*) *Dry Pigment.*—The pigment shall consist of pure para-nitraniline red toner free from any substrate, and shall conform to the following requirements:

Pure organic coloring matter, min., per cent	97.0
Ash, max., per cent	3.0
Moisture and other volatile matter, max., per cent	1.0
Coarse particles (total residue retained on a No. 325 (44-micron) sieve), max., per cent	1.5

(*b*) *Paste in Oil.*—The paste in oil shall be a dispersion of the specified pigment in linseed oil. As received it shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions, without curdling, with linseed oil, turpentine, or volatile petroleum spirits, or any mixture of these substances. The paste shall conform to the following requirements:

Pigment, min., per cent	40.0
Linseed oil, max., per cent	60.0
Moisture and other volatile matter, max., per cent	0.7
Coarse particles and skins (total residue retained on a No. 325 (44-micron) sieve), max., per cent of the dry pigment	2.0

(*c*) The mass color and character of the tint formed by mixture with a white pigment shall be the same as, and the strength not less than, that of a reference sample mutually agreed upon by the purchaser and the seller.

Number of Tests

3. Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1938 to 1939.

ratio of two samples for each 10,000 lb., except that for shipments of less than 10,000 lb. two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

Methods of Testing

4. Tests shall be conducted in accordance with the appropriate methods of the American Society for Testing Materials, where applicable. Test procedures not covered by A.S.T.M. methods shall be mutually agreed upon by the purchaser and the seller.

Standard Specifications for

C.P. TOLUIDINE TONER[1]

A.S.T.M. Designation: D 656 - 43

ADOPTED, 1943.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 656; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover the red pigment commercially known as chemically pure toluidine toner. The pigment may be purchased in the dry form or as a paste in oil.

Composition and Properties

2. (*a*) *Dry Pigment.*—The pigment shall consist of pure toluidine red toner (metanitro-paratoluidine-azo-betanaphthol, with or without a small admixture of a suitable homologue) free of any substrate, and shall conform to the following requirements:

Pure organic coloring matter, min., per cent	98.0
Ash, max., per cent	1.0
Moisture and other volatile matter, max., per cent	1.0
Coarse particles (total residue retained on a No. 325 (44-micron) sieve), max., per cent	1.0
Paranitraniline red	none
Solubility in chloroform	complete

(*b*) *Paste in Oil.*—The paste in oil shall be a dispersion of the specified pigment in linseed oil. As received it shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions, without curdling, with linseed oil, turpentine, or volatile petroleum spirits, or any mixture of these substances. The paste shall conform to the following requirements:

Pigment, min., per cent	40.0
Linseed oil, max., per cent	60.0
Moisture and other volatile matter, max., per cent	0.7
Coarse particles and skins (total residue retained on a No. 325 (44-micron) sieve), max., per cent of the dry pigment	1.5

(*c*) The mass color and character of the tint formed by mixture with a white pigment shall be the same as, and the tinting strength and lightfastness not less than, that of a reference sample mutually agreed upon by the purchaser and the seller.

Number of Tests

3. Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1942 to 1943.

unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10,000 lb., except that for shipments of less than 10,000 lb. two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

Methods of Testing

4. (*a*) *General.*—Tests shall be conducted in accordance with the appropriate methods of the American Society for Testing Materials, where applicable. Test procedures not covered by A.S.T.M. methods shall be mutually agreed upon by the purchaser and the seller.

(*b*) *Paranitraniline Red.*—Place approximately 0.5 g. of the pigment on a clean smooth surface, such as a piece of milk glass. Add a few drops of alcoholic potassium hydroxide solution (22 g. of KOH dissolved in 1000 ml. of ethyl alcohol) to the center of the sample and rub with a glass rod. The presence of paranitraniline red is indicated by a violet color.

(*c*) *Solubility in Chloroform.*—Place about 0.05 g. of pigment in a 50-ml. Nessler tube, add 40 ml. of chloroform (U.S.P.), and warm slightly, stirring with a glass rod. Compare this solution with a similarly prepared solution of a standard sample of known purity. Complete solubility of the pigment in chloroform is indicated if a clear red orange solution is obtained.

NOTE.—Any turbidity indicating an impurity may be conveniently identified by extracting 5 g. of the pigment in a Soxhlet extractor, using chloroform as the solvent. The residue may then be examined and estimated.

Standard Specifications for

ALUMINUM POWDER FOR PAINTS[1]

ALUMINUM BRONZE POWDER

A.S.T.M. Designation: D 266 – 41

ADOPTED, 1931; REVISED, 1939, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 266; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover two types of aluminum powder, commercially known as aluminum bronze powder, for use in paints:

Type A.—Standard fineness, for general paint use.

Type B.—Extra fine, for special uses.

Composition and Properties

2. (*a*) The aluminum powder shall consist of commercially pure aluminum in the form of fine, polished flakes and shall contain no filler or adulterant, such as mica. The powder shall be suitable for making aluminum paint and shall also conform to the following requirements:

Easily extracted fatty and oily matter (polishing lubricant), max., per cent	3.0
Total impurities, other than fatty and oily matter, max., per cent	2.0
Coarse particles, max., per cent:	
Type A; total residue retained on a No. 100 (149-micron) sieve	0.2
Type B; total residue retained on a No. 325 (44-micron) sieve	1.0

(*b*) The powder shall show good "leafing" properties. A paint made from the powder shall be free flowing and shall give a continuous coating at least equal in smoothness, luster, and general appearance to that of a reference sample mutually agreed upon by the purchaser and the seller.

Number of Tests

3. Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10,000 lb., except that for shipments of less than 10,000 lb. two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1927 to 1931, being revised in 1928

Methods of Testing

4. Several of the methods for testing aluminum powder are empirical and the results are affected by the method. The operator shall follow closely the procedures described in the Standard Methods of Sampling and Testing Aluminum Powder and Paste (A.S.T.M. Designation: D 480) of the American Society for Testing Materials.[3]

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for
ALUMINUM PIGMENT PASTE FOR PAINT[1]

A.S.T.M. Designation: D 474 - 41

ADOPTED, 1939; REVISED, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 474; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover two types of aluminum pigment paste for use in paints:

Type A.—Standard fineness, for general paint use.

Type B.—Extra fine "lining," for special finishes.

Composition and Properties

2. (*a*) The aluminum pigment paste shall consist of commercially pure aluminum in the form of fine, polished flakes, combined with a volatile paint thinner and a suitable fatty lubricant to form a paste suitable for use as a paint pigment. It shall contain no fillers or adulterants, such as mica. The paste shall conform to the following requirements:

Nonvolatile matter at 105 to 110 C., min., per cent	64.5
Easily extracted fatty and oily matter (polishing lubricant), max., per cent	4.0
Total impurities, other than fatty and oily matter, max., per cent	1.0
Coarse particles (total residue retained on a No. 325 (44-micron) sieve), max., per cent:	
Type A	1.5
Type B	0.1

NOTE.—All of the above limits are expressed in percentage of the *paste*.

(*b*) The paste shall show good "leafing" properties. A paint made with the paste shall be free flowing and shall give a continuous coating at least equal in smoothness, luster, and general appearance to that of a reference sample mutually agreed upon by the purchaser and the seller.

(*c*) Paste of both types shall meet the stability test described in Section 4. Paste which within six months storage in unopened original packages after shipment is found to be unfit for use will be rejected.

Number of Tests

3. Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10,000 lb., except that for shipments of less than 10,000 lb. two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1938 to 1939.

Methods of Testing

4. Several of the methods for testing aluminum paste are empirical, and the results are affected by the method. The operator shall follow closely the procedures described in the Standard Methods of Sampling and Testing Aluminum Powder and Paste (A.S.T.M. Designation: D 480) of the American Society for Testing Materials.[3]

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for
GOLD BRONZE POWDER[1]

A.S.T.M. Designation: D 267 – 41

ADOPTED, 1931; REVISED, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 267; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover the materials commercially known as gold bronze, pale gold bronze, and rich gold bronze powders.

Composition and Properties

2. (*a*) The bronze powder shall be made from new ingot metals. It shall consist of fine polished flakes with not to exceed 3 per cent of fatty or oily matter (polishing lubricant) to give good "leafing" properties.

(*b*) The residue retained on a No. 100 (149-micron) sieve, using alcohol as the wash liquid, shall not exceed 0.2 per cent.

(*c*) The powder shall have good "leafing" properties. (By "leafing" is understood the property of forming an apparently continuous brilliant film over the entire free surface of a mixture of the powder in a suitable liquid (Note), within 1 min. after cessation of stirring the mixture.) In testing for leafing properties the powder shall be mixed in the proportion of 3 to 4 lb. to a gallon of the liquid. As thus mixed it shall also give a free flowing, smooth, continuous coating.

NOTE.—A suitable liquid is made by mixing spar varnish conforming to the U. S. Federal Specification for Water-Resisting Spar Varnish (No. TT-V-121a) with turpentine conforming to the Standard Specifications for Spirits of Turpentine (A.S.T.M. Designation: D 13) of the American Society for Testing Materials[3] in such proportions that the mixture will have a viscosity of 0.65 to 0.85 poises (*B* to *C* of the Gardner-Holdt scale).

(*d*) The gold bronze powder shall be suitable for making gold bronze paint. It shall match in shade and fineness a reference sample mutually agreed upon by the purchaser and the seller.

Number of Tests

3. Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1927 to 1931, being revised in 1928.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

taken from different packages in the ratio of two samples for each 10,000 lb., except that for shipments of less than 10,000 lb. two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

Methods of Testing

4. Tests shall be conducted in accordance with the appropriate methods of the American Society for Testing Materials, where applicable. Test procedures not covered by A.S.T.M. methods shall be mutually agreed upon by the purchaser and the seller.

Standard Specifications for

ZINC DUST (METALLIC ZINC POWDER)[1]

A.S.T.M. Designation: D 520 – 41

ADOPTED, 1940; REVISED, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 520; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover metallic zinc powder, in the form commercially known as zinc dust, for use as a pigment in paints.

Composition and Properties

2. (*a*) The pigment shall consist substantially of metallic zinc and shall conform to the following requirements:

Metallic zinc, min., per cent	96
Material other than metallic zinc and ZnO, max., per cent	1.5
Moisture and other volatile matter, max., per cent	0.1
Zinc oxide	remainder
Coarse particles, max., per cent:	
Total residue retained on a No. 100 (149-micron) sieve	none
Total residue retained on a No. 325 (44-micron) sieve	4.0

(*b*) In such physical properties as are specified by the purchaser, the pigment shall satisfactorily match a reference sample mutually agreed upon by the purchaser and the seller.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1939 to 1940.

Number of Tests

3. Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10,000 lb., except that for shipments of less than 10,000 lb. two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

Methods of Testing

4. Tests shall be conducted in accordance with the appropriate methods of the American Society for Testing Materials, where applicable. Test procedures not covered by A.S.T.M. methods shall be mutually agreed upon by the purchaser and the seller.

Standard Specifications for

TOXIC INGREDIENTS IN ANTI-FOULING PAINTS[1]

DRY CUPROUS OXIDE, DRY MERCURIC OXIDE

A.S.T.M. Designation: D 277 – 31

ADOPTED, 1931.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 277; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover the toxic ingredients (dry cuprous oxide and dry mercuric oxide) in anti-fouling paints

Composition

2. (*a*) Anti-fouling paints, when mixed ready to apply, shall contain both cuprous oxide and mercuric oxide in the following amounts:

	Cuprous Oxide, min., oz. per U. S. gal.	Mercuric Oxide, min., oz. per U. S. gal.
For general service	14	7
In north temperate waters	25	1.5
In south temperate waters	20	5
In tropical waters	14	14

(*b*) The amount of cuprous oxide material used in the manufacture of anti-fouling paints shall be in proportion to the cuprous oxide content, any cupric oxide present being considered as inert material.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1928 to 1931, being revised in 1931. Editorially revised and rearranged in 1939.

Cuprous Oxide

3. The cuprous oxide shall be a fine powder and shall conform to the following requirements:

Cuprous oxide, min., per cent	96.0
Coarse particles (total residue retained on a No. 325 (44-micron) sieve), max., per cent	2.5

Mercuric Oxide

4. The mercuric oxide shall be a finely powdered red or yellow mercuric oxide conforming to the following requirements:

Chemically combined mercury, calculated as metallic mercury, min., per cent	91.0
Free mercury	none
Ash, max., per cent	0.5
Impurities	practically free from acids and alkalies and from inert organic or inorganic fillers or adulterants.
Coarse particles (total residue retained on a No. 325 (44-micron) sieve), max., per cent	10.0

Methods of Testing

5. The requirements enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials:

(*a*) *Cuprous Oxide.*—Standard Method of Chemical Analysis of Dry Cuprous

Oxide (A.S.T.M. Designation: D 283).[3]

(*b*) *Mercuric Oxide.*—Standard Method of Chemical Analysis of Dry Mercuric Oxide (A.S.T.M. Designation: D 284).[3]

(*c*) *Coarse Particles.*—Procedure for insoluble dry pigments as described in Section 3 of the Standard Methods of Test for Coarse Particles in Pigments, Pastes, and Paints (A.S.T.M. Designation: D 185).[3]

(*d*) *Ash.*—Standard Method of Chemical Analysis of Dry Mercuric Oxide (A.S.T.M. Designation: D 284).[3]

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: K51-1941

Standard Method of Test for
ALKALINITY OR ACIDITY OF PIGMENTS[1]

A.S.T.M. Designation: D 278 – 31

ADOPTED, 1931.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 278; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test covers a procedure for determining the alkalinity or acidity of pigment.

Procedure

2. Shake a 20-g. sample of the dry pigment with 200 ml. of distilled water for 5 min., let settle, filter through a dry filter paper into a dry beaker, discarding the first 10 ml. of the filtrate. Transfer 100 ml. of the clear filtrate to a flask, add 3 drops of methyl orange solution (prepared by dissolving 1 g. of methyl orange in 1 liter of distilled water), and titrate to the end point with H_2SO_4 solution (0.02 *N*) if alkaline, or NaOH or Na_2CO_3 solution (0.02 *N*) if acid.

Calculation

3. Calculate alkalinity to milligrams of NaOH in 1 g. of the sample. Calculate acidity to milligrams of NaOH required to neutralize 1 g. of the sample (1 ml. of exactly 0.02 *N* solution is equivalent to 0.0008 g. of NaOH).

[1] Under the standardization procedure of the Society this method is under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, this method was published as tentative from 1928 to 1931. Editorially revised and rearranged in 1939.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: K32-1941

Standard Methods of Test for
BLEEDING OF PIGMENTS[1]

A.S.T.M. Designation: D 279 – 31

ADOPTED, 1931.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 279; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods cover procedures for determining the "bleeding" of pigments.

Procedure for Dry Pigments to be Used in Cellulose Ester Lacquers

2. Mix a suitable amount of the pigment to be tested with a minimum volume of dibutyl phthalate to give a thick paste when rubbed up with a sharp-edged steel spatula on a glass plate. Stir this paste into a clear cellulose ester lacquer of a composition agreed upon by the buyer and the seller and apply the paint evenly by brushing on a clean, smooth, dry metal panel. Apply the paint to only a part of the panel, leaving a margin of at least 2.5 cm. (1 in.) of unpainted metal around the painted portion. Allow the panel to dry for 30 min. in a well-ventilated room at room temperature (70 to 90 F.). Flow successive coats of white lacquer of a composition agreed upon by the buyer and the seller over the entire surface of the panel until complete hiding is obtained, allowing the panel to dry for 20 min. in a well-ventilated room at room temperature (70 to 90 F.) after applying each coat. Do not apply any white lacquer after the application of the particular coat that causes complete hiding. Let the completed panel dry for 24 hr. and note the color of the two sections of the panel. If the color of the section containing the pigment under test does not match that of the adjoining area containing only the white lacquer, report the pigment as "bleeding."

Procedure for Dry Pigments to be Used in Oil or Oleoresinous Paints and Enamels

3. (*a*) Mix a suitable amount of the material to be tested with a minimum volume of a mixture of the following:

Raw linseed oil[a] ..... 5 parts by volume
Spar varnish[b] ..... 4 parts by volume
Liquid paint drier[c] ..... 1 part by volume

[a] The raw linseed oil shall conform to the Standard Specifications for Raw Linseed Oil (A.S.T.M. Designation: D 234) of the American Society for Testing Materials.[3]
[b] The spar varnish shall conform to the U. S. Federal Specification for Water-Resisting Spar Varnish (No. TT-V-121a).
[c] The liquid paint drier shall conform to the U. S. Federal Specification for Liquid Paint Drier (No. TT-D-651).

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these methods were published as tentative from 1928 to 1931, being revised in 1929. Editorially revised and rearranged in 1939.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(*b*) Rub up the sample prepared in accordance with Paragraph (*a*) with a sharp-edged steel spatula on a glass plate to obtain a uniformly smooth, stiff paste. Thin with turpentine to give a paint of brushing consistency, and apply evenly by brushing on a clean, smooth, dry metal panel. Apply the paint to only a part of the panel, leaving a margin of at least 2.5 cm (1 in.) of unpainted metal around the painted portion. Allow the panel to dry for 18 hr. in a well-ventilated room at room temperature (70 to 90 F.). Brush a coat of Damar varnish (Damar resin cut in turpentine) over the entire surface of the panel. Report any color, other than that of the Damar varnish on the unpainted portion of the panel, as "bleeding."

Procedure for Pastes in Oil

4. Mix a suitable amount of the paste to be tested with sufficient liquid paint drier[c] to give a paint that will dry within 7 hr. Thin the mixture with turpentine to give a paint of brushing consistency. Thoroughly stir the paint, strain it through a No. 100 (149-micron) sieve,[4] and apply by brushing to a clean, smooth, dry metal panel. Apply the paint to only a part of the panel, leaving a margin of at least 2.5 cm. (1 in.) of unpainted metal around the painted portion. Allow the panel to dry (should not require over 7 hr.) in a well-ventilated room at room temperature (70 to 90 F.). After a total of 18 hr. of drying under the above conditions, brush a coat of Damar varnish (Damar resin cut in turpentine) over the entire surface of the panel. Report any color, other than that of the Damar varnish on the unpainted portion of the panel, as "bleeding."

Procedure for Pastes in Japan

5. Mix a suitable amount of the paste to be tested with turpentine to give a paint of brushing consistency. Add the turpentine slowly while thoroughly mixing with a sharp-edged steel spatula or paddle. Thoroughly stir this paint, strain it through a No. 100 (149-micron) sieve,[4] and apply by brushing to a clean, smooth, dry metal panel. Apply the paint to only a part of the panel, leaving a margin of at least 2.5 cm. (1 in.) of unpainted metal around the painted portion. Allow the panel to dry for 5 hr. in a well-ventilated room at room temperature (70 to 90 F.). Brush a coat of Damar varnish (Damar resin cut in turpentine) over the entire surface of the panel. Report any color, other than that of the Damar varnish on the unpainted portion of the panel, as "bleeding."

[4] Detailed requirements for this sieve are given in the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

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AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: K42-1937

Standard Methods of Test for

COARSE PARTICLES IN PIGMENTS, PASTES, AND PAINTS[1]

A.S.T.M. Designation: D 185 – 37

ADOPTED, 1926; REVISED, 1927, 1929, 1937.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 185; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods of test cover procedures for determining the amount of coarse particles in dry pigments and of coarse particles and skins in mixtures of pigments and vehicles.

Apparatus

2. The apparatus shall consist of a 3-in. No. 325 (44-micron) sieve conforming to the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials.[3] A 3-in. No. 325 (44-micron) sieve for comparison purposes should be retained in the laboratory as a reference standard. Whenever a new sieve is secured, a practical test of its accuracy should be made by running on it and on the reference standard sieve a comparison test, using a pigment that has a considerable percentage of coarse particles. A reserve stock of such a pigment should be kept for this purpose.

Procedure for Insoluble Dry Pigments, Except Metallic Aluminum and Bronze Powders

3. (*a*) The sieve shall be dried in an oven at 105 to 110 C., cooled, and then weighed on an analytical balance, the weight being recorded to the nearest milligram.

(*b*) The sample (25 g. for basic carbonate and basic sulfate white leads, 25 g. for red lead and mercuric oxide, 2 g. for black pigments of low specific gravity, 3 g. for Prussian blues and graphite, and 10 g. for all other pigments) of the pigment to be tested shall be weighed on an analytical balance to the nearest milligram. The sieve shall be wet on both sides with alcohol and the sample of pigment shall then be transferred to the sieve and wetted with alcohol.

(*c*) The sieve shall then be held under a tap delivering about 300 to 500 ml. of the wash liquid (water) per minute. By slightly shaking the sieve, the pigment will be rapidly carried through. A soft camel's-hair brush may be used in aiding the operation. If the sieve is held at a slight angle so that the pigment will gradually collect at one edge during

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these methods were published as tentative from 1924 to 1926, being revised in 1926. Editorially revised and rearranged in 1929.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

the washing process, and then rotated, the pigment may be brushed out rapidly, with no risk of clogging the sieve. After the most of the finely divided portion of the pigment has passed through the sieve (from 2 min. to 1 hr., according to the kind of pigment), the sieve shall be placed in an 8-in. porcelain dish containing 250 ml. of the wash liquid (water). The sieve will thus be covered to a depth of about ½ in. The pigment remaining on the sieve shall be brushed with a soft 1-in. camel's-hair brush at the rate of two strokes per second during two periods of 10 sec. each. The sieve shall then be raised from the dish after each 10-sec. period to let the liquid on the sieve run through. The liquid in the dish should be changed after every two brushing periods. This operation shall be continued until the wash liquid (water) passing over the residue and through the sieve is clear and free from solid particles. When the washing appears to be complete, about 200 ml. of the wash liquid (water), after passing over the residue and through the sieve, shall be collected in a clean 400-ml. beaker. The liquid shall be stirred vigorously, and the beaker set on a black surface in the case of white pigments and on a white surface in the case of colored pigments. The washing shall not be considered complete until such a test fails to show any particles collected about the middle of the bottom of the beaker (Note).

(*d*) When the washing is complete the pigment particles adhering to the brush shall be washed back onto the sieve and the water below the sieve wiped off. A few drops of alcohol and then of ether should be added to expedite drying. The sieve shall be dried for 1 hr., cooled, and weighed as described in Paragraph (*a*). The percentage of coarse particles shall then be calculated.

NOTE.—Occasionally, pigments will be found that foam when water is used as the wash liquid. In such instances, during the last washing in the porcelain dish the use of a liquid that breaks down the foaming and is readily miscible with water, such as alcohol, will usually overcome this difficulty.

Procedure for Metallic Aluminum and Bronze Powders

4. In the case of metallic aluminum and bronze powders the procedure described in Section 3 shall be followed except that 5 g. of the material shall be used as a test sample, and denatured alcohol, instead of water, shall be used as the wash liquid.

Procedure for Water-Soluble Pigments, Pastes in Oil, Pastes in Japan, and Mixed Paints.

5. (*a*) The sieve shall be dried, cooled and weighed as described in Section 3 (*a*).

(*b*) The sample to be tested shall contain 10 g. of pigment (25 g. of pigment with white leads and red lead). The weight shall be determined on an analytical balance to the nearest milligram and the sample transferred to a 250-ml. beaker. With dry pigments and pastes 100 ml. of kerosine shall be added to the beaker; with mixed paints and enamels 50 ml. of kerosine. The sieve shall then be wet on both sides with kerosine. The sample and the kerosine in the beaker shall be thoroughly mixed, breaking up all lumps, but not grinding, with the flattened end of a stirring rod. The contents of the beaker shall then be transferred to the sieve, using a wash bottle containing kerosine. When small particles of pigment are retained on the stirring rod or walls of the beaker, they may easily be removed with a camel's-hair brush.

(*c*) The procedure described in Section 3 (*c*) shall then be followed, using kerosine (siphoned from a vessel) instead of water as the wash liquid. The pigment particles adhering to the brush shall then be washed back onto the sieve

and all kerosine shall be washed from the sieve with ether. The sieve shall be dried for 1 hr., cooled, and weighed, as described in Section 3 (*a*). The percentage of coarse particles and skins (in the case of dry pigments, coarse particles) shall then be calculated.

Procedure for Ship-Bottom Paints Containing Resins and Alcohol.

6. In the case of ship-bottom paints containing resins and alcohol the procedure described in Section 5 shall be followed, except that a sample containing 10 g. of pigment shall be used and denatured alcohol, instead of water, shall be used as the wetting medium, for mixing with the paint, and as the wash liquid.

Procedure for Cellulose Ester Lacquers.

7. In the case of cellulose ester lacquers the procedure described in Section 5 shall be followed, except that a sample containing 10 g. of the material shall be used and a mixture of equal parts of ethyl acetate, benzol, and denatured alcohol, instead of water, shall be used as the wetting medium for mixing with the lacquer and as the wash liquid.

Standard Method of Test for

RELATIVE DRY HIDING POWER OF WHITE PIGMENTS IN A LINSEED OIL VEHICLE[1]

A.S.T.M. Designation: D 406 - 39

ADOPTED, 1939.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 406; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test is intended for determining the relative dry hiding power of a white pigment in a linseed oil vehicle and is only to be used for a comparison of standard and sample of the same type of white pigment. An optional procedure is included which permits a quantitative determination of the relative dry hiding powers of two pigments of the same type.

Definition

2. Relative dry hiding power of a white pigment is the ability of that pigment to impart hiding power to a paint prepared in the manner specified below. It is quantitatively expressed as the relative dry hiding power of this paint[3] in terms of a similar paint prepared from a standard pigment.

Apparatus

3. The apparatus shall consist of the following:

(*a*) *Balance.*—Regulation laboratory balance with capacity of 100 g. and sensitivity of 0.1 g.

(*b*) *Syringe.*—Syringe of 10-ml. capacity.

(*c*) *Paint Brush.*—A good grade paint brush, 1½ in. in width.

(*d*) *Container.*—A ½-pt. can or a 250-ml. beaker.

(*e*) *Test Surface.*—A smooth-surfaced paper chart having adjacent black and white areas and coated with a suitable varnish or lacquer so as to render the surface impervious and resistant to paint liquids. The black areas of the board shall have a diffuse reflectance of less than 1 per cent. The white areas shall have a diffuse reflectance of 75 per cent or greater. The white areas of the boards used for the standard paint and the sample shall not differ in brightness by more than 1 per cent. The chart shall have a test area of not less than 1 sq. ft. and any suitable regular geometri-

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, this method was published as tentative from 1935 to 1939, being revised in 1937 and 1939.

[3] See the definition of relative dry hiding power of a paint in the Standard Method of Test for Relative Dry Hiding Power of Paints (A.S.T.M. Designation: D 344), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

cal design of the contrasting areas may be used.

(*f*) *Illumination.*—A reasonable intensity of diffused light, preferably northern skylight or a source approximating northern skylight.

(*g*) *Burette.*—A standard 25-ml. burette, graduated in 0.1 ml. (stopcock controlled). The tip of the burette shall be ground optically flat.

(*h*) *Glass Muller.*—Standard type glass muller with a grinding face of from $2\frac{3}{4}$ to 3 in. in diameter. The face shall be free from blowholes and other imperfections, and kept sharp by slightly grinding with No. 303 optical emery or its equivalent and turpentine. The edge should be beveled.

(*i*) *Rubbing Surface.*—Lithographers' stone or ground plate glass slab, the surface of which is kept sharp by slightly grinding with No. 303 optical emery or its equivalent and turpentine.

(*j*) *Spatula.*—A flexible spatula having a 6-in. blade for mixing the oil and pigment.

Materials

4. The following materials will be required:

(*a*) *Standard Pigment.*—A standard white pigment of the same type as that to be tested mutually agreed upon by the purchaser and the seller.

(*b*) *Vehicle.*—The vehicle shall be prepared from the following two types of linseed oil, combining 50 per cent of each by weight to which shall be added 1 ml. of a cobalt naphthenate drier (metal content approximately 6 per cent) to each 100 g.

(1) Heat-bodied, alkaline, refined linseed oil, having a viscosity of E to F on the Gardner-Holdt scale[4] and an acid number of 1 to 5.

(2) Light, boiled linseed oil, having a viscosity of B on the Gardner-Holdt scale,[4] an acid number of 2 to 5, and color not darker than No. 11 on the 1933 Gardner Color Standard.[5]

Procedure

5. The relative dry hiding power of the white pigment shall be determined as follows:

(*a*) The standard white pigment and vehicle shall be first mulled to a stiff paste and then reduced to a paint of reasonably good brushing properties. Quantities of pigment and vehicle for both operations shall be mutually agreed upon by the purchaser and the seller. The quantities of pigment and vehicle used should produce paints of reasonably good brushing properties from both standard and sample. Table I is intended to serve only as a guide.

TABLE I.—SUGGESTED QUANTITIES OF PIGMENT AND VEHICLE.

Pigment	Pigment, g.	Oil for Mulling, ml.	Oil for Reducing Single Mulling, ml.
Zinc oxide	17.8	5.6	6.3
Leaded zinc oxide	16.3	3.5	8.7
Antimony oxide	25.3	5.3	5.3
Basic carbonate white lead	25.5	4.3	7.0
Basic carbonate white lead (high oil)	25.5	6.2	6.5
Basic sulfate white lead	25.5	5.0	6.0
Titanium dioxide	11.6	6.6	5.6
Titanated lithopone	17.9	7.5	3.4
Titanium barium (30 per cent)	18.9	5.9	4.6
Titanium calcium (low oil)	13.9	6.1	4.4
Titanium calcium (high oil)	12.4	8.1	3.0
Titanium magnesium	12.9	7.6	3.8
Lithopone (low-oil absorption)	13.9	3.8	8.0
Lithopone (high-oil absorption)	10.5	4.4	8.2
Zinc sulfide barium	11.8	4.2	8.0
Zinc sulfide magnesium	10.6	5.0	6.9
Zinc sulfide	10.7	4.4	7.9

(*b*) The amount of standard white pigment shall be weighed accurately and the corresponding quantity of vehicle measured out, taking care to allow sufficient time for complete drainage of the burette (approximately 5 min.). The

[4] Gardner's Laboratory Manual, Ninth Edition, May 1939, p. 216.

[5] Gardner's Laboratory Manual, Ninth Edition, May, 1939, p. 72.

materials shall be placed on the rubbing slab and worked into a paste with the spatula. Then the paste shall be rubbed up with the glass muller, using a stroke approximately 6 in. in width and from 12 to 15 in. in length. In counting rubs, one stroke up and one stroke back shall be considered to be one rub. The muller shall be allowed to travel up one side and back the other side, twisting the muller slightly at the top and bottom of each half stroke to help work in the pigment. After each 25 rubs with the muller, the pigment shall be "picked up" with the spatula by scraping the face of the muller and gathering the paste on the slab into a daub. The mulling shall be continued until the paste is given 100 rubs. This paste shall then be thinned with the additional vehicle and transferred to the container.

(*c*) The sample of white pigment to be tested shall be treated in the same manner as the standard, as described in Paragraphs (*a*) and (*b*).

(*d*) Brushouts of these paints shall be prepared as described in Paragraph (*e*) at a reasonable spreading rate mutually agreed upon by the purchaser and the seller.

(*e*) The paint prepared from the standard pigment shall be well stirred and the brush dipped into the paint and worked out on a suitable surface. The desired quantity of paint shall be taken up in the syringe, and the syringe and wet brush weighed in the empty container. The paint shall then be spread over the surface of the chart, the syringe returned to the empty container, and the paint brushed out uniformly over the test surface, care being taken to cover only the test area. The empty syringe, wet brush, and container shall again be weighed. The loss of weight represents the weight of paint applied to the test surface. A test chart shall then be prepared from the paint made with the sample pigment in the same manner and at the same spreading rate. Both painted test surfaces shall then be set aside to dry.

(*f*) After the test surfaces are thoroughly dry, they shall be placed side by side against a flat surface. The standard and sample brushouts shall be viewed in juxtaposition under a reasonable intensity of diffused light, preferably northern skylight or a source approximating northern skylight. The observer should view the brushouts from a distance of 5 to 10 ft.

Relative Dry Hiding Power

6. If the apparent contrast existing on the brushout of the sample of white pigment is adjudged equal to or less than that existing on the brushout of the standard white pigment, the sample shall be said to be equal to or better than the standard pigment in relative dry hiding power as defined in Section 2.

Optional Procedure

7. (*a*) In many cases it is not sufficient to say that the sample pigment is better or poorer than the standard, and the amount of difference must be determined quantitatively. The optional procedure described in the following Paragraphs (*b*) and (*c*) is recommended for determining quantitative differences in hiding power. It may be used in place of the procedure outlined in Section 5.

(*b*) The paint prepared from the sample of white pigment shall be applied to a brushout board as described in Section 5 (*e*) at the spreading rate mutually agreed upon. A series of brushouts shall be made from the paint prepared from the standard white pigment. The difference in spreading rate between successive steps in this series should be approximately 15 per cent. The range of spreading rate covered by

this series should be sufficient to give contrasts above and below that of the brushout of the sample pigment (estimated from the probable difference in relative dry hiding power, if known) or should cover the entire range of spreading rate convenient to apply with the standard paint. The painted test surfaces shall be set aside to dry.

(*c*) Under the conditions of illumination and viewing, as described in Section 5 (*f*), the brushout of the sample paint shall be compared with a series of brushouts of the standard paint, numbered, beginning with one, in order of decreasing spreading rate for convenience of handling. It will be found to be equal in contrast to one brushout of the standard or to be intermediate in contrast between two adjacent standards in the series of standard brushouts. If the sample is found to be between two adjacent standards in contrast, the sample shall be viewed against these standards with the higher and lower standard on either side. For purposes of grading, the contrast interval between the two standards shall be considered as 10 arbitrary units. The position of the sample shall be estimated on this arbitrary scale. Thus, if the sample appears equally distant in contrast from either standard, it will be graded 5 on the arbitrary scale. If nearer the standard of less contrast, it will be graded 6, 7, 8, or 9 as the case might be. The grading shall be recorded in terms of the number of the standard brushout of higher spreading rate followed by a decimal point and the grading of the arbitrary scale. In making this grading it is important that the sample be graded between the two standards with the standard of lesser contrast first on the right and then on the left. The two gradings thus obtained shall be averaged to give the final grading figure.

Calculation

8. The hiding power of the sample of white pigment shall be calculated as follows:

$$\text{Hiding power} = \frac{(W_1 - W_2)\frac{X}{10} + W_2}{W_3} \times 100$$

where:

W_1 = weight of paint on the brushout of the heavier grading standard,

W_2 = weight of paint on the brushout of the lighter grading standard,

W_3 = weight of paint on the brushout of the sample pigment, and

X = contrast grading (decimal portion of the recorded grading).

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BY THE AMERICAN STANDARDS ASSOCIATION
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Standard Method of Test for

HYGROSCOPIC MOISTURE (AND OTHER MATTER VOLATILE UNDER THE TEST CONDITIONS) IN PIGMENTS[1]

A.S.T.M. Designation: D 280 – 33

ADOPTED, 1931; REVISED, 1933.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 280; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test covers procedures for determining hygroscopic moisture (and other matter volatile under the test conditions) in pigments.

Method A. For Pigments that Do Not Decompose at 110 C.

Apparatus

2. The apparatus shall consist of the following:

(*a*) A wide-mouth cylindrical glass weighing bottle (flat form, about 30 mm. in height and about 70 mm. in diameter), provided with a ground-in glass stopper. Or, an aluminum moisture dish (about 90 mm. in diameter and about 50 mm. in depth) with a tightly-fitting cover.

(*b*) An oven in which a temperature of from 105 to 110 C. can be maintained.

(*c*) An analytical balance.

Procedure

3. The procedure shall be carried out as follows:

Weigh accurately the glass weighing bottle and stopper or the aluminum moisture dish with cover. Place a sample of from 3 to 5 g. of the pigment in the clean, dry weighing bottle or in the clean, dry aluminum moisture dish. Insert the stopper (or cover) and weigh. Subtract the weight of the vessel from the total weight to obtain the weight of sample used in the test. Remove the stopper (or cover) and place it and the bottle (or dish) containing the sample in an oven that has been previously heated to 105 to 110 C.; heat for 2 hr. at a temperature of 105 to 110 C. Replace the stopper (or cover), cool in a desiccator, and weigh. Calculate the total loss in weight to percentage of moisture and other volatile matter.

Method B. For Pigments That Decompose at 110 C.

Apparatus

4. The apparatus shall consist of the following:

(*a*) A glass weighing bottle as described in Section 2 (*a*).

(*b*) An open-tube manometer made of glass tubing 6 mm. in diameter, filled

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, this method was published as tentative from 1923 to 1931. Editorially revised and rearranged in 1939.

with mercury to approximately 86 cm., fitted with rubber pressure tubing attached to a T-joint leading to the desiccator and the pump (Note 1).

(*c*) A glass desiccator, having a hole at the side or in the cover, constructed with heavy walls to withstand a vacuum of one atmosphere. The hole at the side shall be closed with a one-hole rubber stopper carrying a glass tube with a rubber tube connection and a pinchcock or with a glass stopcock ground to fit the tubulature.

(*d*) An oil vacuum pump with a motor to give a vacuum of not more than 3 mm.[3]

(*e*) An analytical balance.

Procedure

5. The procedure shall be carried out as follows:

Weigh accurately the glass weighing bottle and stopper. Place a sample of from 1 to 3 g. of the pigment in the clean, dry weighing bottle, insert the stopper and weigh. Subtract the weight of the vessel from the total weight to obtain the weight of sample used in the test. Remove the stopper and place it and the bottle containing the sample in the desiccator containing *fresh*, magnesium perchlorate trihydrate.[4] Close the desiccator, attach to the pump, and *gradually* evacuate until the manometer indicates that the vacuum, which should not be greater than 3 mm., is constant (Note 2). Close the pinchcock or stopcock, stop the pump, and let stand at room temperature (70 to 90 F.) for 24 hr. Admit air that has been dried by passage through *fresh* magnesium perchlorate trihydrate slowly to the desiccator by means of the pinchcock or stopcock, remove the cover, quickly replace the stopper in the weighing bottle, and weight. Repeat the evacuations and weighings until the loss in weight does not exceed 0.5 mg. in 24 hr. Calculate the total loss in weight to percentage of moisture and other volatile matter (Note 3).

EXPLANATORY NOTES

NOTE 1.—The difference in levels of the mercury in the manometer when the system is in operation, subtracted from the barometer reading taken at the same time, gives the vacuum of the system in millimeters of mercury. The difference between the barometer and the manometer reading should not exceed 3 mm.

NOTE 2.—Caution should be used in evacuating glass desiccators. The vacuum should be applied gradually. Desiccators will maintain a vacuum for a greater length of time when a thin film of a suitable stopcock lubricant[5] is applied to the ground surfaces. Desiccators when evacuated for the first time should be wrapped in towels or covered by other means to prevent possible injury to the operator.

NOTE 3.—This is an arbitrary method and the details should be strictly followed. The determination of the true hygroscopic moisture content of very finely divided pigments is very difficult, if not impossible in some cases.

[3] The "Hyvac" oil pump is satisfactory.

[4] H. H. Willard and G. Frederick Smith, "The Preparation and Properties of Magnesium Perchlorate and Its Use as a Drying Agent," *Journal*, Am. Chemical Soc., Vol. 44, pp. 2255–59 (1922). The compound is sold under the trade name "Dehydrite."

[5] Martin Shepherd and P. G. Ledig, "Rubber Stopcock Lubricants for High Vacuum and Other Uses," *Industrial and Engineering Chemistry*, Vol. 19, p. 1059 (1927).

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: K54-1941

Standard Method of Test for
OIL ABSORPTION OF PIGMENTS[1]

A.S.T.M. Designation: D 281 – 31

ADOPTED, 1931.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 281; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test covers a procedure for determining the oil absorption of pigments.

Procedure

2. Weigh exactly 1 g., or any multiple thereof (Note 1), of the thoroughly mixed and air-dried pigment, and place upon a glass plate or marble slab. Weigh accurately a dropping bottle (Note 2) fitted with a ground-in pipette and rubber bulb and containing some clear, well-settled, raw linseed oil (free from foots) having an acid value (Note 3) of from 1 to 3 (Note 4). Add the linseed oil gradually, drop by drop (by means of the pipette) to the pigment, and after the addition of each drop thoroughly incorporate the oil with the pigment by rubbing up with a sharp-edged steel spatula. The test is complete when exactly enough oil has been incorporated with the pigment to produce a very stiff, putty-like paste, which does not break or separate. Accurately weigh the bottle and oil (Note 2) and determine by difference the weight of oil used.

NOTE 1.—The weight of sample depends upon the specific gravity, fineness, and other characteristics of the pigment. For example, if 20 g. is taken for white lead, about 1 g. is sufficient for carbon black. In any event the size of sample should be large enough so that at least 1 g. of oil is required.

NOTE 2.—Instead of weighing the linseed oil in the dropping bottle, it may be added drop by drop from an accurate burette. The number of milliliters of linseed oil used is then multiplied by the specific gravity of the oil in order to obtain the corresponding weight of the oil in grams.

NOTE 3.—Care should be taken that the linseed oil used in comparative or simultaneous tests has the same acid value.

NOTE 4.—While raw linseed oil is specified, other liquids (such as refined oil) may be used by mutual agreement.

Calculation

3. From the weights of oil and pigment used in the test, calculate the number of pounds of oil required to exactly "wet" 100 lb. of pigment.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, this method was published as tentative from 1928 to 1931, being revised in 1931. Editorially revised and rearranged in 1939.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: K41-1939

Standard Methods of Test for
SPECIFIC GRAVITY OF PIGMENTS[1]

A.S.T.M. Designation: D 153 - 39

ADOPTED, 1924; REVISED, 1927, 1939.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 153; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods of test cover procedures for determining the specific gravity of pigments.

METHOD A. FOR ROUTINE TESTING OF SEVERAL SAMPLES SIMULTANEOUSLY

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Pycnometer.*—A pycnometer, as shown in Fig. 1 (*a*) or (*b*), having a capacity of 50 ml.

(*b*) *Water Bath.*—A water bath consisting of a vessel filled with sufficient water to permit of only a very gradual rise in temperature and equipped with a stirring device, preferably air-blown.

(*c*) *Manometer.*—An open-tube manometer, see *f*, Fig. 3, made of glass tubing 6 mm. in diameter, filled with mercury to approximately 86 cm., fitted with rubber pressure tubing attached to a T-joint leading to the desiccator and the pump.

NOTE.—The difference in levels of the mercury in the manometer when the system is in operation, subtracted from the barometer reading taken at the same time, gives the vacuum of the system in millimeters of mercury. The difference between the barometer and the manometer readings should not exceed 3 mm.

(*d*) *Desiccator.*—A glass desiccator, having a hole at the side, constructed with heavy walls to withstand a vacuum of one atmosphere.

(*e*) *Vacuum Pumps.*—A laboratory water vacuum pump to expel the greater portion of air in the desiccator.

An oil vacuum pump[3] with a motor to give a vacuum of not more than 3 mm.

(*f*) *Weighing Bottle.*—A weighing bottle with cork. The neck shall be small enough to fit inside the neck of the pycnometer. This latter requirement is essential, since small quantities of pigment easily adhere to the ground-glass joint of the pycnometer.

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these methods were published as tentative from 1923 to 1924.

[3] The "Hyvac" oil pump is satisfactory. This pump has a displacement of approximately 200 cu. in. per min. or 7 cu. ft. per hr. at a speed of 900 rpm. It will evacuate a flask of 1-liter capacity to a vacuum of 1 mm. in 2 min.

(*g*) *Thermometer.*—A thermometer having a range of from 0 to 60 C. graduated in 0.1 C.

Standardization of Pycnometers

3. The pycnometer shall be filled with freshly-boiled distilled water and brought to a temperature of 15.6 C. It shall be dried and weighed as specified in Section 4 (*f*). It shall then be cleaned, weighed, and dried. It shall then be filled with the kerosine to be used in making the test and again brought to a temperature of 15.6 C., and dried and weighed in the same manner as before. The specific gravity of the kerosine shall be calculated as follows:

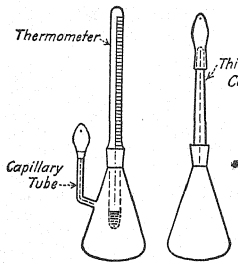


FIG. 1.—Pycnometers.

$$\text{Sp. gr., } 15.6/15.6 \text{ C.} = \frac{\text{wt. of kerosine}}{\text{wt. of water}}$$

Procedure

4. (*a*) *Drying.*—The pigment shall be dried in an oven at 105 C. for 2 hr.

NOTE.—It is preferable to use an electric oven for this purpose.

(*b*) *Weighing.*—A sample of the pigment shall be weighed, by difference, in the weighing bottle. For black, blue, and lake pigments of light specific gravity, about 1 g. should be used; for inert crystalline pigments, about 4 g.; for opaque white pigments, 7 to 10 g.; and for red lead, from 15 to 20 g. should be used.

NOTE.—Due to the hygroscopic nature of some of the pigments it is necessary to use a weighing bottle fitted with a cork stopper.

(*c*) *Number of Samples.*—It has been found convenient to run six samples at one time, the desiccator specified being of the proper size to accommodate this number.

(*d*) *Transferring to Pycnometer.*—Sufficient kerosine shall be poured into the pycnometer to form a ¼-in. layer in the bottom and a quantity of pigment from the weighing bottle shall be added reaching approximately three-fourths of the distance to the kerosine level. The kerosine shall always cover the pigment. The sample shall be stirred with a polished round-bottom glass rod until completely covered by the kerosine, more kerosine being added from the wash bottle if necessary. The rod shall be washed with kerosine.

(*e*) *Removal of Occluded Air.*—The pycnometers shall be placed in the desiccator, which shall then be closed and attached to the water pump until the greater part of the air is expelled from the system. This takes from 5 to 10 min. The system shall then be closed with a pinchcock and the desiccator attached to the oil pump for the removal of the small amounts of air given off at the low pressures obtainable with the oil pump. The manometer is used to indicate whether the oil pump is giving the proper vacuum. When the manometer indicates that the vacuum, which should not be greater than 3 mm., is constant, the oil pump may be cut off for short periods, precaution being taken that the vacuum does not change materially due to leakage. It will be noticed that bubbles of air come from the pigments very rapidly at

first and that this action gradually decreases and finally stops altogether. The time required for complete removal of air varies from 30 min. to 2 hr., depending upon the nature of the pigment. When no more bubbles can be seen, it is assumed that all the occluded air has been given off and that the pigment is thoroughly wet with kerosine. Air may then be slowly admitted to the desiccator by means of the pinchcock.

(*f*) *Filling and Bringing to Temperature.*—The pycnometer shall be taken from the desiccator and filled with kerosine, care being taken to add a sufficient quantity to prevent the formation of air bubbles when placement of the thermometer is made. The thermometer shall be placed in the water bath, which shall be cooled with ice to a temperature between 10 and 13 C. The pycnometer shall be placed in the bath and be permitted to come to constant temperature. The pycnometer thermometer or capillary tube shall then be inserted. Enough warm water shall be added to the bath to raise the temperature suddenly to about 14.5 C. in order to expand the kerosine and prevent it from creeping down the capillary and admitting a small amount of air. The bath shall be allowed to come to a temperature of 15.6 C. The capillary tube shall be wiped with filter paper and the cap put on. All temperatures on the thermometer in the bath shall be read, but not on the thermometer in the pycnometer. The pycnometer shall be removed from the bath and dried. It shall then be allowed to stand for 30 min. to enable it to come to room temperature, and weighed.

NOTE.—It is advisable to allow the pycnometer to stand approximately the same time before each weighing so as to compensate for slight errors due to evaporation at the joints.

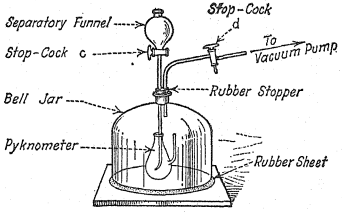


FIG. 2.

Calculations

5. The specific gravity shall be calculated as follows:

$$\text{Sp. gr. of pigment} = \frac{P \times S}{(P + K) - F}$$

where:

K = weight of the bottle filled with kerosine only,
P = weight of pigment used,
F = final weight of the bottle with pigment and kerosine, and
S = specific gravity of the kerosine used.

Precautions

6. Before a new desiccator is used for the first time, it shall be wrapped in a towel and tested under the vacuum to be used, great care being exercised in handling the desiccator when the vacuum is on, as any sudden jar may cause it to collapse.

METHOD B. FOR TESTS REQUIRING HIGHEST ACCURACY

Apparatus

7. The apparatus shall consist of the following:

(*a*) *Pycnometer.*—A pycnometer as shown in Fig. 1 (*a*) or (*b*), having a capacity of 50 ml.

(*b*) *Water Bath.*—A water bath consisting of a vessel filled with sufficient water to permit of only a very gradual rise in temperature and equipped with a stirring device, preferably air-blown.

(*c*) *Bell Jar.*—A glass bell jar with a two-hole rubber stopper. Into one hole of the stopper shall be fitted a separatory funnel with a well-ground stopcock *c*, Fig. 2, and with the lower tube extending into the pycnometer (just below the opening of the side arm in Fig. 1 (*a*)). Into the other hole of the stopper shall be fitted a glass tube with a well-ground 3-way stopcock *d*, Fig. 2, the tube connecting with the vacuum pump *e*, Fig. 3. The bell jar shall rest on a sheet of rubber, cemented or vulcanized to a glass or iron plate. With stopcock *c* closed and stopcock *d* opened to the pump, the system shall withstand and maintain a vacuum.

NOTE.—A suitable desiccator may be substituted for the bell jar.

(*d*) *Thermometer.*—A thermometer having a range of from 0 to 60 C., graduated in 0.1 C.

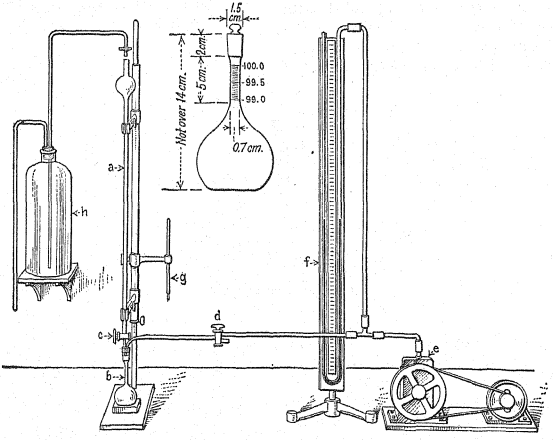


FIG. 3.—Apparatus Assembly for Determining Specific Gravity of Pigments.

(*e*) *Vacuum Pump.*—A high vacuum pump *e*, Fig. 3.

(*f*) *Manometer.*—An open-tube manometer *f*, Fig. 3, as described in Section 1 (*c*).

(*g*) *Bottle.*—A storage bottle *h*, Fig. 3, for kerosine or other wetting liquid.

Procedure

8. The pycnometer containing the weighed sample of dried pigment shall be placed under the bell jar. Stopcocks

c and *d* shall be closed, the vacuum pump started, and then stopcock *d* gradually opened to the pump. When the air has all been removed, with a vacuum of 1 mm. maintained, the separatory funnel

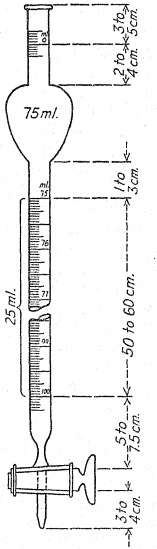


FIG. 4.—Glass Burette.

Burette: Geissler, straight; glass stopcock, ground accurately.

Total capacity	100 ml.
Capacity of bulb	0 to 75 ml.
Graduated	75 to 100 ml.
Subdivisions	0.05 ml.
Rate of outflow	about 2 min.
Permissible Variations:	
Total capacity	±0.10 ml.
Graduated portion	±0.03 ml.

Markings on graduations shall be in conformity with the National Bureau of Standards *Circular No. 9.*

shall be filled with kerosine, stopcock *d* closed, and at once stopcock *c* shall be gradually opened, adding sufficient kerosine to completely cover the pigment. The pump shall be stopped and the suction at *d* released. Finally, the pycnometer shall be filled with kerosine and the test completed as described in Section 4 (*f*) and Section 5 under method *A*.

NOTE 1.—Before a new bell jar (or desiccator) is used for the first time, it shall be tested under a vacuum as described in Section 6.

NOTE 2.—Stopcock *c* must be well ground and should be lubricated with castor oil or glycerol. See precautions in Section 15 under method *C* concerning the necessity of testing the system for leaks before making a determination.

NOTE 3.—With certain pigments that are not wetted well with kerosine, the substitution of turpentine has been found very efficient. However, when turpentine, or any other liquid having a high evaporation rate, is used, a pycnometer of type shown in Fig. 1 (*a*) or (*b*) is not satisfactory, on account of losses around the ground-glass joints. When using such liquids as turpentine, the bottle (as a pycnometer) and apparatus as described in Section 9 under method *C* should be used, except that instead of attempting to measure an accurate volume of the liquid from the burette, the bottle (plus the sample plus the wetting liquid) is finally weighed as in method *B*.

METHOD C. FOR RAPID AND ACCURATE TESTING OF SINGLE SAMPLES

Apparatus

9. The apparatus shall consist of the following (see Fig. 3):

(*a*) *Burette.*—A 100-ml. burette with a 75-ml. bulb in the upper part and the lower part (25 ml.) graduated to 0.05 ml. (see Fig. 4).

(*b*) *Flask.*—A special 100-ml. graduated flask, *b*, Fig. 3, with ground-glass stopper. The flask shall be thick enough to withstand and maintain a vacuum and should weigh between 50 and 60 g. The neck of the flask should be graduated in 0.05 ml. between the 99 and 100-ml. marks. The dimensions of the flask are shown in Fig. 3.

(*c*) *Stopcocks.*—A tightly ground stopcock *c*, in the burette *a*.

A 3-way stopcock *d*, connecting with the vacuum pump *e*.

(*d*) *Vacuum Pump.*—A high-vacuum

pump[3] *e*, of the type requiring no preliminary or "backing" pump.

(*e*) *Manometer.*—An open-tube manometer *f*, Fig. 3, as described in Section 2 (*c*).

NOTE.—The difference in levels of the mercury in the manometer when the system is in operation, subtracted from the barometer reading taken at the same time, gives the vacuum of the system in millimeters of mercury. The difference between the barometer and the manometer readings should not exceed 1 mm.

(*g*) *Thermometer.*—A thermometer *g*, having a range of from 0 to 60 C. graduated in 0.1 C.

(*h*) *Bottle.*—A storage bottle *h*, for kerosine or other wetting liquid.

Standardization of Apparatus

10. The flask shall be connected to the burette and pump by means of a two-hole rubber stopper. The system shall be evacuated with stopcock *c* closed until the pump maintains a vacuum of 1 mm. in the flask (this requires only a few minutes). The 3-way stopcock *d* shall be closed for 30 sec., and again opened to the pump. There should be no appreciable change in the mercury levels in the manometer, indicating that the system beyond stopcock *d* is tight. With the vacuum still maintained, the burette shall be filled from the top with kerosine, the level adjusted to the zero mark with a piece of capillary tubing. Stopcock *d* shall now be closed and then stopcock *c* carefully opened, admitting about 75 ml. of kerosine into the flask. Stopcock *d* shall be opened to the air, thus releasing the vacuum in the flask, and the flask shall be filled with kerosine to a definite mark on the neck. The burette shall be read, calling this reading *K* (the volume of the flask).

Procedure

11. The flask shall then be cleaned with ether, dried, and weighed. A glass counterpoise having the same superficial area and treated the same way may be placed in the opposite pan in the balance. A quantity of the dry pigment to be tested shall be transferred to the flask by means of a clean, dry, glass funnel whose stem reaches to the bottom of the bulb. A piece of stiff nickel wire is convenient in pushing the powder down the stem. The bulb of the flask should be nearly filled with the sample which, however, should occupy a volume of less than 25 ml. after all air is expelled. Greater accuracy may be obtained with a large sample than with a small one. The inside stem as well as the entire outside of the flask should be wiped with a clean piece of dry, lintless cloth. The flask and pigment shall then be weighed and the weight of pigment calculated by deducting the weight of the empty flask. With the burette clean and dry, but with the stopcocks well lubricated (castor oil or glycerol), the flask shall be attached to the evacuating system as shown in Fig. 3. After closing stopcocks *c* and *d*, the pump shall be started and stopcock *d* carefully opened to the pump. Evacuation shall be continued until the pump maintains a vacuum of 1 mm. in the flask, or until all the air is expelled from the system. The burette shall then be filled from the top as described in Section 10, stopcock *d* closed, stopcock *c* gradually opened and kerosine added until the pigment is covered. The flask shall be tapped gently to dislodge any air bubbles. The pump should be stopped, stopcock *d* opened to the air, and the flask filled up to the same mark as was obtained in determining its volume. The volume of kerosine required may be designated *X*. The

height of the liquid in the burette shall be read to the nearest estimated 0.01 ml.

NOTE.—The removal of all air can not be stressed too greatly, as the presence of air will cause low results.

Calculations

12. The specific gravity shall be calculated as follows:

$$\text{Sp. gr. of pigment} = \frac{S}{K - X}$$

where:

S = weight of pigment used,
K = volume of kerosine required to fill the flask when empty, and
X = volume of kerosine required to fill the flask when the pigment is present.

Temperature

13. Since the specific gravity of a pigment is only slightly affected by temperature, such variations as occur under normal conditions in a room would not materially affect the results. Care shall be taken, however, that the temperature of the liquid after transferring to the flask is approximately the same as it was when in the burette.

Wetting Medium

14. While kerosine has been found to be a good wetting medium, any liquid which does not have a high evaporation rate may be used. The liquids are interchangeable, as no constants on them need be determined. Hence, a pigment containing a dye which is slightly soluble in kerosine could be run with another liquid in the same apparatus without special standardization for that liquid. Operators are cautioned against the use of water as it causes considerable frothing with certain pigments.

Precautions

15. (*a*) Care shall be taken that the burette stopcock is well ground in order to prevent leakage of kerosine. Castor oil is suggested as the stopcock lubricant.

(*b*) Since in determining both K and X, the tip of the burette and bore of the stopcock plug are empty, no correction is needed; stopcock *c*, however, should be so well ground that under a vacuum of 1 mm. for 30 min. no leakage of kerosine takes place. The usual sources of error are failure to remove all the air from the pigment and leaks in the system. The minimum amount of rubber tubing should be used anywhere in the system, and wherever this is used the joints between rubber and glass should be coated with a melted mixture of beeswax and rosin.

(*c*) In cleaning the flask of kerosine only, a rinsing two or three times with ether followed by dry air (dried over sulfuric acid and calcium chloride) is sufficient. When pigment is also present, both pigment and kerosine should be taken together and then emptied. This should be followed with ether until no more pigment is removed. Some filter pulp (macerated filter paper) and water (with or without some glass beads) should be added and shaken vigorously. Repetition may be necessary. The flask should then be rinsed with distilled water and either dried in an oven or rinsed with alcohol and ether followed by dry air. In determining specific gravity by this method, there is no reason why the flask, if made of resistant glass,[4] cannot be heated, followed by cooling during evacuation if such heating has no effect on the sample.

[4] Pyrex glass is very satisfactory for this purpose.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: K56-1941

Standard Method of Test for

TINTING STRENGTH OF WHITE PIGMENTS[1]

A.S.T.M. Designation: D 332 – 36

ADOPTED, 1936.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 332; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test is intended for determining the tinting strength of white pigments or white pigment pastes and is only to be used for a comparison of the sample and a reference standard of the same type of white pigment.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Balance.*—Regulation laboratory balance, sensitive to at least 1 mg.

(*b*) *Burettes.*—Standard 1-ml. burettes, graduated in 0.1 ml., (stopcock controlled) or the apparatus known as the Brown burette, which consists of a small burette as just described but with a small reservoir. The tips of all burettes shall be ground optically flat.

(*c*) *Glass Mullers.*—Standard type glass mullers with a grinding face of from $2\frac{3}{4}$ to 3 in. in diameter. The face shall be free from blowholes and other imperfections, and kept sharp by lightly grinding with No. 303 optical emery or its equivalent and turpentine. The edge should be beveled.

(*d*) *Rubbing Surface.*—Lithographers' stones or ground plate glass slabs, the surface of which is kept sharp by lightly grinding with No. 303 optical emery or its equivalent and turpentine.

(*e*) *Glass or Celluloid Sheets.*—Sheets of flat glass or flexible celluloid for use in weighing out the oils and pastes.

(*f*) *Spatula.*—A flexible spatula having a 3-in. blade for mixing the oil and pigment.

(*g*) *Panels.*—Bright tin or clear glass panels for use in observing color matches.

(*h*) *Scraper.*—A French scraper or body glazing scraper for smoothing down the color daubs placed on the glass or tin panels. The scraper shall have a blade that is about 3 or 4 in. in width with a good straight edge.

Materials

3. The following materials will be required:

(*a*) *Reference Standard.*—A standard white pigment or white pigment paste

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, this method was published as tentative from 1931 to 1936, being revised in 1934 and 1935. Editorially revised and rearranged in 1939.

agreed upon by the seller and the purchaser.

(*b*) *Tinting Material.*—Either ultramarine blue or a very fine, low-strength, uniform black tinting material prepared from finest lampblack (Note 1). If desired, the tinting material, either ultramarine blue or black, may be prepared in the form of an oil paste (Note 2) ground fine and placed in small collapsible tubes.

NOTE 1.—It is advisable to prepare such a pigment from the finest lampblack and the finest precipitated calcium carbonate by intimate mixing of one part of the dry lampblack and six parts of the dry calcium carbonate until a uniform mixture is obtained which will give check results with several duplicate samples of a standard white pigment.

NOTE 2.—Recommended formulas for such a paste using ultramarine blue are as follows:

No. 1.
- 140 lb. Ultramarine blue
- 50 lb. Poppy oil
- 3 lb. Calcium stearate
- 2 lb. Castile soap
- ½ lb. Turpentine

No. 2.
- 70 lb. Ultramarine blue
- 27 lb. Refined linseed oil
- 3 lb. 4-hr. cooked linseed oil (560 F.)

(*c*) *Oil.*—White refined linseed oil with an acid number of approximately 4. The use of bodied oils or varnish may be essential in some cases.

Procedure for White Pigments

4. The tinting strength of white pigments shall be determined as follows:

(*a*) Weigh accurately 2 g. of the reference standard white pigment and the amount of ultramarine blue or lampblack and measure the amount of oil as indicated in Table I.

(*b*) Use sheets of flat glass or flexible celluloid in weighing out the pigments, oils and pastes. For regular control work, the use of the burette is recommended for measuring the oil used, instead of weighing, because of the simplicity, speed, and reasonable accuracy of the burette. Be sure to allow sufficient time for the burette to drain to its true level. In case of doubt or dispute over any tint or in the case of viscous oils, all portions shall be weighed. If desired, the tinting material may be added in the form of a paste, and in this case the weighed quantity of paste shall have the amount of tinting material specified in the above table and shall be added to the mulled sample on the rubbing slab and thoroughly mixed with the spatula until no streaking is noticeable. Place the materials on the rubbing surface and work them into a paste with the spatula, then rub up the paste with the glass muller, using a stroke 3 to 4 in. in width and from 12 to 15 in. in length. In counting the rubs, one stroke up and one stroke back is considered one rub. Allow the muller to travel up one side and back the other side, twisting the muller slightly at the top and bottom of each half stroke to help work in the pigment. After each 25 rubs with the muller, "pick up" the pigment with the spatula by scraping the face of the muller and gathering the paste on the slab into a daub. Continue the mulling until the paste is given 100 rubs.

TABLE I

Pigment	Weight of Pigment, g.	Amount of Oil, ml.	Weight of Tinting Material, g.	
			Ultramarine Blue	Lampblack
White lead	2	0.4	0.2	0.04
Zinc oxide	2	0.6	0.2	0.04
Zinc oxide (35 per cent leaded)	2	0.4	0.2	0.04
Lithopone	2	0.5	0.2	0.04
Extra strength lithopone	2	0.5	0.2	0.04
Zinc sulfide	2	0.6	0.4	0.08
Titanium barium pigment	2	0.5	0.2	0.04
Titanium calcium pigment	2	0.5	0.2	0.04
Titanium dioxide	2	0.9	0.4	0.08

(*c*) Treat 2 g. of the sample of white pigment in exactly the same manner as prescribed in Paragraph (*a*) for the reference standard, using the same

amount of tinting material and oil. Give the sample 100 rubs. Place the sample and reference standard in juxtaposition on a bright tin or clear glass panel. Make the daub of each about 1 in. in width and 2 in. in length and draw the scraper lightly over the pastes to even off the ridge and so place both daubs on an even plane. If glass is used, the rub-outs may be observed either from the top or through the glass panel.

(*d*) If the sample differs appreciably from the reference standard in tint, other pastes of the reference standard white pigment may be prepared, using various amounts of the tinting material to give a series of standards. The sample of white pigment shall then be compared with the series of rub-outs of the reference standard pigment, the quality of tint being judged by placing the rub-outs in juxtaposition on the tin or glass panel as in the procedure described in Paragraph (*c*).

Calculation

5. The tinting strength of the sample shall be calculated as follows:

$$\text{Tinting strength of pigment} = \frac{A}{B} \times T$$

where:

A = weight in grams of tinting material used with reference standard to give equality of tint,

B = weight in grams of tinting material used with sample, and

T = empirical tinting strength value given to the reference standard.

Optional Procedure for White Pigments

6. (*a*) The method described in Sections 4 and 5 is satisfactory for routine testing of samples of white pigments of the kind which do not vary greatly in tinting strength. Variations in the brightness or consistency of the tinted pastes used for matching cause variations in the relative tinting strength of the samples being tested. When comparing similar pigments which do not vary greatly in tinting strength, these errors are negligible, but when comparing pigments which do vary greatly in tinting strength, the comparison should best be made with tinted pastes of standard brightness and standard consistency. In the following method all comparisons shall be made with pastes that have a brightness or light reflection of 20 per cent and equal consistency.

(*b*) Weigh accurately 2 g. of the reference standard white pigment and a sufficient amount of the blue (or black) tinting material to produce a paste which will have a 20 per cent light reflection (Note 1) when mixed with oil. Place these materials on the rubbing surface and add sufficient oil to produce a paste of medium consistency (Note 2). Mull the sample as described in Section 4 (*b*) until the paste is given 100 rubs.

NOTE 1.—The light reflection may be measured by the Marten's photometer or other suitable instrument or by comparison with a paste known to have the specified light reflection.

NOTE 2.—This consistency shall be such that the paste can just be thrown from the spatula by a gentle whipping motion of the hand or by the tapping of the spatula blade with the index finger.

(*c*) Treat 2 g. of the sample of white pigment in exactly the same manner as described in Paragraph (*b*) for the reference standard, using sufficient tinting material to produce a paste which will exactly match the standard. Use sufficient oil with the sample of white pigment to produce a paste having approximately the same consistency as the reference standard. Give the sample 100 rubs as described in Section 4 (*b*). It will be necessary to prepare several 2-g. portions of the sample of white pigment using various amounts of the

tinting material until the color of the reference standard is exactly matched. Judge the color match as described in Section 4 (*c*).

Calculation (Optional Procedure)

7. The tinting strength of the unknown sample shall be calculated from the following formula:

$$\text{Tinting strength of pigment} = \frac{C}{D} \times T$$

where:

C = weight in grams of tinting material used with sample to give equality of tint,

D = weight in grams of tinting material used with reference standard, and

T = empirical tinting strength value given to the reference standard.

Procedure for Pastes

8. When the sample is in paste form, the tinting material should also preferably be in the form of an oil paste. The weighed quantity of tinting material in paste form shall be added to the weighed sample of white pigment paste on the rubbing slab and thoroughly mixed with the spatula until no streaking is noticeable. The tinting strength shall then be determined in accordance with the procedure described in Section 4 (*c*) and the calculation made as in Section 5. If desired, the optional procedure in Section 6 (*c*) may be used and the calculation made as in Section 7.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: K57-1941

Standard Method of Test for

MASS COLOR AND TINTING STRENGTH OF COLOR PIGMENTS[1]

A.S.T.M. Designation: D 387 – 36

ADOPTED, 1936.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 387; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test covers procedures for determining the mass color and tinting strength of the materials generally known as dry colors, either in the form of dry pigments or in the form of pastes in oil or japan. They do not apply to the white pigments.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Balance.*—Regulation laboratory balance, sensitive to at least 1 mg.

(*b*) *Burettes.*—Standard 1-ml. burettes, graduated in 0.1 ml., (stopcock controlled) or the apparatus known as the Brown burette, which consists of a small burette as just described but with a small reservoir. The tips of all burettes shall be ground optically flat.

(*c*) *Glass Mullers.*—Standard type glass mullers with a grinding face of from 2¾ to 3 in. in diameter. The face shall be free from blowholes and other imperfections, and kept sharp by lightly grinding with No. 303 optical emery or its equivalent and turpentine. The edge should be beveled.

(*d*) *Rubbing Surface.*—Lithographers' stones or ground plate glass slabs, the surface of which is kept sharp by lightly grinding with No. 303 optical emery or its equivalent and turpentine. To guard against contamination, different slabs or stones should be available for red, yellow, blue, green, and black pigments.

(*e*) *Glass or Celluloid Sheets.*—Sheets of flat glass or flexible celluloid for use in weighing out the samples of dry colors, oils, and pastes.

(*f*) *Spatula.*—A flexible spatula having a 3-in. blade for mixing the oil and pigment.

(*g*) *Panels.*—Bright tin or clear glass panels for use in observing color matches.

(*h*) *Scraper.*—A French scraper or body glazing scraper for smoothing down the color daubs placed on the glass or tin panels. The scraper shall have a

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, this method was published as tentative from 1934 to 1936, being revised in 1935. Editorially revised in 1939.

blade that is about 3 or 4 in. in width with a good straight edge.

Materials

3. The following materials will be required:

(*a*) *Reference Standard.*—A reference standard pigment (dry color) or paste color in oil or japan agreed upon by the seller and the purchaser.

(*b*) *Reduction Paste.*—Zinc oxide oil paste for the tinting strength test (Note).

NOTE.—Recommended formulas for such a paste are as follows:

No. 1[a]	300 lb. Green seal zinc oxide 64 lb. Poppy oil 2 lb. Calcium stearate 1 lb. Turpentine
No. 2[b]	90 lb. Green seal zinc oxide 10 lb. Refined linseed oil 1 lb. 4-hr. cooked linseed oil (560 F.)
No. 3[b]	100 lb. Green seal zinc oxide 25 lb. Heat bodied oil (of "O" Litho varnish consistency)

[a] This formula yields a short paste, handy to use and which has little tendency to settle. The paste should be ground fine and placed in small collapsible tubes.

[b] This formula yields a paste that is slightly stringier than No. 1 and settles very little. New oil pastes should be prepared at least once each month. The paste should be ground fine and placed in small collapsible tubes.

(*c*) *Oil.*—White refined linseed oil with an acid number of approximately 4. In special cases, other oils may be essential, for example, earth colors may require raw oil, or pigments for printing inks require transparent lithographic varnish. Such special oils should be arranged for by the seller and the purchaser.

MASS COLOR

Procedure

4. Mass color shall be determined as follows:

(*a*) Carefully weigh the sample of pigment to be tested and counterbalance with the reference standard pigment. For regular control work, the use of the burette is recommended for measuring the oil used, instead of weighing, because of the simplicity, speed, and reasonable accuracy of the burette. In case of doubt and dispute over any color, or in the case of viscous oils, all portions shall be weighed. If oil is to be weighed, add oil to the dry samples accurately. Transfer the oil and pigment to the rubbing slab. If oil is to be measured, transfer the weighed samples of the pigments to the slab and add oil by dropping (slowly) from the burette, working up the paste with the small spatula. Be sure to allow sufficient time for the burette to drain to its true level. When all the dry pigment is worked up by means of the spatula, rub up the paste with the glass muller. Use a stroke from 3 to 4 in. in width and from 12 to 15 in. in length. In counting the rubs given a color, one stroke up and one stroke back is considered one rub. Allow the muller to travel up one side and back the other side, twisting the muller slightly at the top and bottom of each half stroke to help work in the pigment. After the number of rubs with the muller, as specified in the following table, depending on the type of pigment, "pick up" the paste with the spatula by scraping the face of the muller and gathering the paste on the slab into a daub. Continue the mulling until the paste is given the specified number of rubs:

Total Number of Rubs Required for Pigment	Pick-up Required
300	after each 50 rubs
200	after each 40 rubs
150	after each 30 rubs
100	after each 25 rubs
80	after each 20 rubs
60	after each 15 rubs

(*b*) When the mulling is completed, place the sample and the reference standard in juxtaposition on the bright tin or clear glass panel. Make the daub of each about 1 in. in width and 2 in. in length and draw the scraper lightly over the pastes to even off the ridges and so present both daubs on an even plane.

Mass tone should be judged immediately. If glass is used observe the rub-outs from the top and not through the glass panel.

NOTE.—Where higher precision is required than that afforded by visual examination or where it is desirable to determine the color characteristics of the material in terms of fundamental physical units, this may be done by use of the Standard Method of Test for Spectral Characteristics and Color of Objects and Materials (A.S.T.M. Designation: D 307) of the American Society for Testing Materials.[3]

TINTING STRENGTH OF COLOR PIGMENTS

Procedure

5. The tinting strength of color pigments shall be determined as follows:

(*a*) Weigh the required amount of the mulled color paste from the slab and counterbalance with the reference standard pigment. Then add the correct amount of the "reduction paste" (zinc oxide oil paste) to the sample and the reference standard. Thoroughly mix each of the pastes on the flat glass or flexible celluloid sheets and transfer samples to the slab, mixing them thoroughly with the spatula only until no more streaking is noticeable.

(*b*) Place the sample and reference standard in juxtaposition on the bright tin or clear glass panel. Make the daub of each at least 1 in. in width and about 2 in. in length and draw the scraper lightly over the pastes to even off the ridge and so present both daubs on an even plane. Judge the color immediately. If glass is used, observe the rub-outs from the top and not through the glass panel.

NOTE 1.—The following pigment rub-out scheme is outlined for the convenience of the operator. Because of the difference in oil absorption of similar pigments made by different manufacturers, the pigment rub-out scheme can necessarily serve only as a guide in the work.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

PIGMENT RUB-OUT SCHEME

Color or Class	Amount of Color, g.	Volume of Oil, ml.	Mulls or Rubs	Reduction grams of reduction paste to grams of mulled paste
c.p. Iron blues	1.0	0.80	300	5 to 0.1
c.p. Light and medium para toners	0.5	0.80	200	5 to 0.1
c.p. Dark para toners	0.6	0.80	300	5 to 0.1
10 per cent Para toners on whitting	1.0	0.50	60	2 to 0.1
c.p. Toluidine	0.5	0.50	150	5 to 0.1
c.p. Light chrome green	1.0	0.40	100	2 to 0.1
c.p. Medium chrome green	1.0	0.50	150	2 to 0.1
c.p. Deep chrome green	1.0	0.60	200	2 to 0.1
25 per cent Grinder's green, light	1.0	0.30	60	2 to 0.1
25 per cent Grinder's green, medium	1.0	0.40	60	2 to 0.1
25 per cent Grinder's green, deep	1.0	0.40	80	2 to 0.1
c.p Chromium oxide	2.0	0.40	60	2 to 0.1
c.p. Primrose chrome yellow	1.0	0.20	60	2 to 0.1
c.p. Light chrome yellow	1.0	0.50	60	2 to 0.1
c.p. Medium chrome yellow	1.0	0.30	60	2 to 0.1
c.p. Dark chrome yellow	2.0	0.40	60	2 to 0.1
c.p. Zinc yellow	1.0	0.40	60	2 to 0.1
English vermillion	2.0	0.50	100	2 to 0.1
Ultramarine blue	1.0	0.50	150	2 to 0.1
Cobalt blue	1.0	0.50	150	2 to 0.1
High strength carbon black	0.3	2.80	150	5 to 0.1
Bone black	1.0	0.80	100	2 to 0.1
Lampblack	0.3	1.00	100	5 to 0.1
Carbon black	0.3	1.30	150	5 to 0.1
Ferrite yellow	0.5	0.30	100	2 to 0.1
French yellow ochre	1.0	0.55	100	2 to 0.1
Light red oxide	1.0	0.70	100	2 to 0.1
Indian, Spanish, Venetian, Persian Gulf oxides and metallic brown	1.0	0.40	100	2 to 0.1
Raw and burnt sienna	1.0	0.60	100	2 to 0.1
Raw and burnt umber	1.0	0.80	100	2 to 0.1

NOTE 2.—In many cases, the paint manufacturer producing industrial, freight car, marine, barn, or anticorrosive paints is interested in wetting the pigment with the vehicle. Such methods of grinding in oil do not reduce the particle size or develop additional tinctorial value of the pigment. The covering power of such paints is therefore dependent upon the degree to which the color manufacturer develops the pigment used in such products. The muller tests do not determine the true value of colors for this purpose. The grinding action of the muller develops the tinctorial value and hiding power of the pigment to a much greater extent than when the pigment is ground in oil on a loosely set stone mill. Comparative muller and stone grinding tests show that fifty mulls with no weight on the muller are equivalent to a loose grind on a paint mill in which the pigment is thoroughly wetted. It is recommended that this test be used in determining the true value of these pigments for paints wherein the pigment receives little or no grinding in the paint mill.

NOTE 3.—For the most exact determination of mass color and ultimate tinting strength, it is recommended that the following procedure be employed: The comparison of all samples shall be made at the same or equal consistency and this consistency shall be such that the paste can just be thrown from the spatula by a gentle whipping motion of the hand or by the tapping of the spatula blade with the index finger.

NOTE 4.—To determine the percentage strength of the sample of the color in terms of the reference standard, varying amounts of the mulled paste of the standard color are mixed with the same amount of reduction paste to give a series of reference standards. The sample is compared to determine the reference standard it matches. The tinting strength of the sample is calculated as follows: The ratio of the amount of the reference standard color used to give equality of tint, to the amount of sample used, multiplied by 100, is the tinting strength of the sample.

TINTING STRENGTH OF PASTES

Procedure

6. When the sample is in the paste form as a color in oil or color in japan, the tinting strength shall be determined in accordance with the procedure described in Section 4 (*b*) and continuing in Section 5 (*a*) and (*b*).

Standard Methods of

SAMPLING AND TESTING ALUMINUM POWDER AND PASTE[1]

A.S.T.M. Designation: D 480 - 44

ADOPTED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 480; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods cover the procedure for the sampling, qualitative analysis, and physical testing of aluminum powder and aluminum paste for paint.

Sampling

2. (*a*) Subject to mutual agreement between the seller and the purchaser, one package shall be taken as a representative sample from each lot of 1000 packages or fraction thereof. Whenever possible, the original unopened package of the material to be tested shall be sent to the laboratory. If it is not possible to send the original package, the inspector representing the purchaser shall thoroughly mix the contents of the container and take a sample of not less than 0.5 lb. (0.227 kg.). This sample shall be placed in a clean, dry, metal or glass container which shall be nearly filled, then closed with a tight cover, sealed, marked, and sent to the laboratory for test.

(*b*) When requested, a duplicate sample may be taken from the same package and delivered to the seller, and the inspector may take a third sample to hold for test in case of disagreement.

Qualitative Analysis

3. (*a*) *Aluminum Powder.*—Wash approximately 0.5 g. of the aluminum powder with ether to remove most of the fatty or oily matter. Place the powder in a beaker and add 50 ml. of diluted HCl (1:1). If an appreciable amount of insoluble matter remains after the addition of the acid, the powder does not conform to the specifications. Dilute a portion of the HCl solution and saturate with H_2S. No appreciable precipitate should form with pure aluminum powder. To another portion of the HCl solution add a slight excess of NH_4OH (sp. gr. 0.90) and note the characteristic precipitate of $Al(OH)_3$ (Note).

(*b*) *Aluminum Paste.*—Volatilize the solvent from an approximately 0.5-g. sample of the aluminum paste, wash the residue with ether to remove most of the fatty and oily matter. Treat the sample in accordance with the procedure described in Paragraph (*a*) (Note).

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these methods were published as tentative from 1938 to 1944, being revised in 1941 and 1942.

NOTE—In general these qualitative tests will suffice, but if doubt exists as to the purity of the powder or paste, a complete qualitative analysis shall be made. If the qualitative analysis indicates impurities in excess of the amount permitted, the total impurities shall be determined in accordance with the Tentative Methods of Chemical Analysis of Aluminum and Aluminum Alloys (A.S.T.M. Designation: E 34) of the American Society for Testing Materials.[3]

Leafing Properties

4. (*a*) *Apparatus.*—The apparatus shall consist of the following:

(*1*) *Spatula.*—A polished steel spatula, having the following dimensions:

Length of blade.....not less than 5.5 in.
Width of blade..... 0.540 ± 0.002 in.
Thickness of blade:
1 cm. from tip..... 0.013 ± 0.003 in.
10 cm. from tip..... 0.038 ± 0.004 in.

(*2*) *Test Tube.*—A test tube 6 in. in length by $\frac{3}{4}$ in. in diameter.

(*3*) *Glass Cylinder.*—A glass cylinder 8 in. in length and $1\frac{1}{2}$ to 2 in. in inside diameter. A cork stopper shall be used and the spatula shall be attached to the cork so as to hang vertically in the cylinder with the tip about 1 in. from the bottom.

(*b*) *Reagents.*—The following reagents will be required:

(*1*) *Coumarone-Indene Resin* having the following properties:

Melting point..... 127 to 137 C.
Specific gravity..... 1.07 to 1.09
Acid number, max..... 0.5

(*2*) *Petroleum Spirits* conforming to the Standard Specifications for Petroleum Spirits (Mineral Spirits) (A.S.T.M. Designation: D 235) of the American Society for Testing Materials,[4] and in addition having the following properties:

Specific gravity at 60 F..... 0.793 to 0.795
Surface tension at 70 F., min. 27.0 dynes per cm.

[3] A.S.T.M. Methods of Chemical Analysis of Metals (1943).

[4] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(*c*) *Preparation of Leaf-Testing Vehicle.*—The resin shall be ground to pass a No. 100 (149-micron) sieve. A solution shall be prepared by dissolving 30 g. of the resin in 100 ml. of petroleum spirits, while heating gently. The specific gravity of the solution shall be between 0.875 and 0.882 at 60 F. It shall be allowed to settle and the clear portion retained for use.

(*d*) *Weight of Sample.*—Unless otherwise specified, the weight of sample shall be as follows:

Classification	A.S.T.M. Designation[a]	Type[b]	Weight of Sample g.
Aluminum powder	D 266	*A*	2
		B	1
Aluminum paste	D 474	*A*	1.5
		B	1.5

[a] These designations refer to the following specifications of the American Society for Testing Materials:
Standard Specifications for Aluminum Powder for Paints (Aluminum Bronze Powder) (A.S.T.M. Designation: D 266),[4] and
Standard Specifications for Aluminum Pigment Paste for Paint (A.S.T.M. Designation: D 474).[4]

[b] Type *A* aluminum pigment is known commercially as "standard fineness" and type *B* as "extra fine lining."

(*e*) *Procedure* (Note).—Make the test at a room temperature of 25 ± 2 C. Place 5 ml. of the leaf testing vehicle in the glass cylinder and cover with a watch glass, then transfer 25 ml. of the vehicle to a clean container. Weigh the required sample of aluminum powder or paste to the nearest 0.01 g. and transfer to a small dish. Add about 2.5 ml. of the 25 ml. quantity of vehicle and mix to a stiff paste with a spatula or small stiff brush. Add approximately 5 ml. more of the vehicle and stir to a smooth mixture, then add the remainder of the vehicle and continue stirring to obtain complete dispersion. Immediately transfer enough of the mixture to the test tube to give a depth of about 11 cm. when the specified spatula is immersed.

Any bubbles on the surface of the liquid may be removed by dipping and withdrawing the end of the spatula a few times. Dip the spatula to the bottom of the mixture and rotate it gently through an arc of about 90 deg. for 10 sec. Formation of bubbles shall be avoided. Withdraw the spatula at a uniform rate of not less than 5 cm. per sec. The spatula should not touch the wall of the test tube above the liquid level. Use a stop watch to time the speed of withdrawal. Immediately suspend the spatula vertically in the glass cylinder and allow to remain for 3 min. Measure the height of immersion and the height of complete leafing on both sides of the spatula, measuring to the bottom of the meniscus in each case.

Clean the spatula by rinsing it in petroleum spirits and wipe dry with a clean cloth. Stir the mixture in the test tube and repeat the determination. Make five determinations in all.

(*f*) *Calculation and Report.*—(1) Calculate the leafing value as follows:

$$\text{Leafing value} = \frac{\text{height of complete leafing}}{\text{height of immersion}} \times 100$$

(2) Report the average of the five determinations as the leafing value of the pigment (Note). The limit of accuracy of this test is plus or minus 5 per cent of the leafing value. Report the weight of sample taken for the leafing test.

NOTE.—The leafing test conditions specified are selected arbitrarily, and even though the numerical leafing value obtained by this test appears to be low, the pigment may give substantially perfect leafing under the conditions of practical application in a paint.

Brushing, Smoothness, Luster, and General Appearance

5. (*a*) *Aluminum Powder.*—Compare the sample of aluminum powder to be tested with a sample mutually agreed upon by the purchaser and the seller. Prepare separate mixtures of each of the samples, using the same vehicle, in accordance with the procedure described in the test for leafing properties in Section 4. Allow the mixtures to stand for 2 hr. after the first stirring. Then stir each thoroughly again and apply by brushing to a clean, smooth glass or metal surface and also to white index cards of good quality. Allow the coatings to dry in a nearly vertical position, and by visual inspection compare the smoothness, luster, and general appearance of each coating.

(*b*) *Aluminum Paste.*—Compare the sample of aluminum paste to be tested with a sample mutually agreed upon by the purchaser and the seller. Prepare separate mixtures of each of the samples, using the same vehicle, in accordance with the procedure described in the test for leafing properties of aluminum powder in Section 4, using 10 g. of type A paste and 6 g. of type B paste. Allow the mixtures to stand for 10 min. after the first stirring. Then stir each thoroughly again and apply by brushing to a clean, smooth glass or metal surface and also to white index cards of good quality. Allow the coatings to dry in a nearly vertical position, and by visual inspection compare the smoothness, luster, and general appearance of each coating.

Coarse Particles

6. (*a*) *Aluminum Powder.*—Fill two straight-wall containers approximately 12 in. in diameter to a depth of not less than 4 in. with petroleum spirits, and fill a third similar container to a depth of not less than 3 in. with acetone. The petroleum spirits used for the test shall conform to the Standard Specifications for Petroleum Spirits (Mineral Spirits) (A.S.T.M. Designa-

tion: D 235),[4] and the acetone shall conform to the Standard Specifications for Acetone (A.S.T.M. Designation: D 329) of the American Society for Testing Materials.[4] Weigh 5 g. of the sample, transfer to a 250-ml. beaker, and add in small portions a total of about 150 ml. of petroleum spirits, mixing *thoroughly* to a uniform consistency after each addition. Clamp a standard sieve[5] 8 in. in diameter just above the level of the petroleum spirits in the first container and pour the sample on the sieve, using a No. 100 (149-micron) sieve for type A powder and a No. 325 (44-micron) sieve for type B powder. Rinse the beaker with clear petroleum spirits and transfer the rinsings to the sieve. Holding the sieve at a slight angle, shake it backward and forward, working the sieve alternately just under and just above the level of the liquid. It is important that the material be thoroughly sieved in each container. After the bulk of the sample has passed through the sieve (time required usually about 2 min.) repeat the procedure in the second container for about 2 min. When it is evident that practically none of the residue on the sieve is passing through it, repeat the procedure in the acetone container for about 2 to 3 min., until nothing more passes through the sieve. Rinse the sides of the sieve with a small stream of acetone, and collect the residue at one side of the sieve. Transfer the residue to a dried and weighed aluminum cup or small evaporating dish with a small stream of acetone, using not less than 15 to 25 ml. Place the aluminum cup or dish in an oven, evaporate to dryness at 105 to 110 C., and continue heating at this temperature for 15 min. (*Caution:* Do not close the door of the oven while evaporating the acetone, as there is danger of explosion or flash of acetone vapor.) Cool the dish and weigh. Calculate the residue retained on the sieve from the increase in weight as follows:

$$\text{Residue retained on sieve, per cent} = \frac{\text{wt. of residue}}{\text{wt. of sample}} \times 100$$

NOTE 1.—The end point of the sieving is easily observed after a few tests have been made. The total time for actual sieving should not be more than 15 min. No brush or other aid shall be used during the sieving. It is necessary that all lumps of pigment be thoroughly dispersed in the petroleum spirits while in the beaker before transferring to the sieve.

NOTE 2.—Results tend to increase as the acetone becomes fouled with particles of powder. It is therefore important to filter the acetone regularly.

NOTE 3.—A correction factor shall be applied in the case of sieves used in the test that have not been certified by the National Bureau of Standards. Sieves having a correction factor above 1.2 or below 0.8 should not be used. The correction factor may be calculated as follows: Determine the coarse particles of a sample on a master sieve certified by the National Bureau of Standards following the procedure described in Paragraph (*a*). Sieve the same sample on the unknown sieve by the same procedure. Assuming that the residue retained on the certified sieve is 1.0 per cent, and that the residue on the unknown sieve is 1.2 per cent, then the correction factor is 0.833.

(*b*) *Aluminum Paste.*—Determine the percentage of coarse particles in aluminum paste in accordance with the procedure described in Paragraph (*a*), using a No. 325 (44-micron) sieve for all types of paste.

Easily Extracted Fatty and Oily Matter

7. (*a*) *Aluminum Powder.*—Weigh accurately approximately 2 g. of the sample, transfer to a 200-ml. volumetric flask and add about 50 ml. of redistilled acetone. The acetone used for this test shall conform to the Standard

[5] Detailed requirements for these sieves are given in the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Specifications for Acetone (A.S.T.M. Designation: D 329) of the American Society for Testing Materials.[4] Heat the mixture to boiling, agitating occasionally, cool, fill to the mark with acetone and mix. Allow the mixture to settle, draw off about half of the supernatant liquid, and filter through dry paper. Discard the first 10 ml. of the filtrate, transfer 100 ml. of the clear filtrate to a weighed dish, evaporate the acetone at a temperature not above 75 C., heat the dish for 15 min. in an oven at 105 to 110 C., cool, weigh, and calculate the percentage of fatty and oily matter.

NOTE.—This is an arbitrary method and the details of the test procedure shall be strictly followed.

(*b*) *Aluminum Paste.*—Determine the percentage of easily extracted fatty and oily matter in aluminum paste in accordance with the procedure described in Paragraph (*a*).

Stability

8. *Aluminum Paste.*—Add 1 ml. of water to a ¼-pt. tin-plated container approximately 2½ in. in diameter and 2 in. in height, provided with a double-friction type seat cover (Note 1). Place the cover loosely on the container and heat in a low-temperature oven until the water is vaporized, remove from the oven, and cool so that the water will condense on the surface of the can. Weigh 100 g. of the paste and place in the container, with a minimum disturbance of the droplets of water. By means of a spatula, break up the paste so as to provide maximum exposure to the moisture. Place the cover on the can and seal tightly. Heat in a thermostatically-controlled oven at 45 ± 0.5 C. for a period of 24 hr. Mix the paste which has been heated in the presence of moisture, with varnish in the proportions of 10 g. of type *A* paste, or 6 g. of type *B* paste, with 40 ml. of varnish (Note 2). The resulting paint, when brushed on metal or glass panels and allowed to dry in a vertical position, shall present the brilliant, lustrous surface which is characteristic of an acceptable aluminum paint. The finish shall be practically equivalent to the finish obtained from a mixture prepared with a sample of the same paste without the addition of water and heating.

NOTE 1.—It is important that the size of the container used shall be as specified.

NOTE 2.—A varnish conforming to the Federal Specification for Mixing Varnish for Aluminum Paint (TT-V-81a) is suitable for this purpose.

Nonvolatile Matter

9. *Aluminum Paste.*—Weigh 5 g. of the paste into a tared evaporating dish. Heat in an oven at a temperature of 105 to 110 C. for 3 hr., cool, and weigh. Calculate the percentage of nonvolatile matter in the sample from the loss in weight, as follows:

$$\text{Nonvolatile matter, per cent} = \frac{\text{wt. of residue}}{\text{wt. of sample}} \times 100$$

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: K55-1941

Standard Method of Test for

ACETONE EXTRACT IN DRY LAMPBLACK AND DRY BONE BLACK[1]

A.S.T.M. Designation: D 305 – 31

ADOPTED, 1931.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 305; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test covers the routine determination of acetone extract in lampblack and bone black (Note 1).

NOTE 1.—This is an arbitrary method and the details should be followed strictly. It is practically impossible to remove all matter soluble in acetone from lampblack. (See Goodwin and Park, *Industrial and Engineering Chemistry*, Vol. 20, No. 6, p. 621 (1928).)

Procedure

2. Weigh a sample of about 2 g., transfer it to a 200-ml. volumetric flask, and add about 50 ml. of acetone (Note 2). Heat the solution to boiling (Note 3), cool, dilute to the mark with acetone, and mix thoroughly. Allow to settle, draw off about 120 ml. of the supernatant liquid, and filter it through a dry filter paper in a dry funnel, receiving the filtrate in a dry vessel. Discard the first 10 ml. of the filtrate, and transfer exactly 100 ml. of the clear subsequent filtrate to a dry, weighed dish. Evaporate this filtrate at a temperature not exceeding 75 C. After evaporation, heat the dish with the residue for 30 min. in an oven at 100 to 105 C., cool, and weigh. Run a blank determination with an equal volume of the acetone (Note 2), and correct the final weight accordingly. Calculate the percentage of acetone extract.

NOTE 2.—The acetone should be redistilled before using.

NOTE 3.—The first boiling and the evaporation may be carried out with proper regulation on a steam bath, but an electric hot plate or heater is preferable.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, this method was published as tentative from 1929 to 1931. Editorially revised and rearranged in 1939.

Standard Method of

CHEMICAL ANALYSIS OF DRY CUPROUS OXIDE[1]

A.S.T.M. Designation: D 283 – 39

ADOPTED, 1929; REVISED, 1933, 1936, 1939.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 283; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method describes procedures for the chemical analysis of dry cuprous oxide pigment.

Treatment of Sample

2. Dry pigments, if lumpy or not finely ground, shall be ground to a fine powder and thoroughly mixed (Note 1). Large samples may be thoroughly mixed and a representative portion taken and powdered if lumpy or not finely ground. The sample in all cases shall be thoroughly mixed before taking portions for analysis. All samples shall be preserved in stoppered bottles or containers (Note 2). Commercial copper oxides appear to segregate rather easily. Therefore, the thorough mixing of the sample to insure homogeneity is an extremely important factor in obtaining concordant results.

NOTE 1.—It is very important that the sample be thoroughly mixed. Some samples of cuprous oxide are not homogeneous, and for this reason are likely to give trouble when an attempt is made to obtain concordant results. By placing a few grams of a sample on a sheet of white paper, and drawing it out with a spatula, it is frequently found that the sample contains coarse particles of black scale, along with small balls of bright red cuprous oxide. Thus it may be necessary to grind such a product thoroughly in a mortar and pestle, in order to make it homogeneous. It is possible that during this operation a slight oxidation may take place.

NOTE 2.—Caution should be used in protecting the sample from oxidation.

TOTAL COPPER

Procedure

3. Weigh accurately a dry sample of from 0.15 to 0.2 g. of the material, transfer it to a 250-ml. beaker, add 10 to 15 ml. of HCl (sp. gr. 1.19) and 1 to 2 ml. of HNO_3 (sp. gr. 1.42), and boil for several minutes. Add 10 ml. of H_2SO_4 (sp. gr. 1.84) and heat until copious fumes of sulfuric acid are evolved. Cool, add cautiously, while stirring, 50 ml. of cold water, and boil to dissolve the sulfates. Cool, filter off any insoluble matter (SiO_2, $PbSO_4$, etc.), and wash the filter and residue until free from copper with a cold H_2SO_4 solution (1:99), catching the filtrate and wash-

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, this method was published as tentative from 1928 to 1929.

ings in a 250-ml. beaker, keeping the total volume of the solution down to about 150 ml. Place in the solution a piece of clean, pure sheet aluminum having a minimum purity of 99.80 per cent and containing a maximum of 0.02 per cent copper. The sheet aluminum is convenient to use in the form of a cylinder about 3.5 to 4 cm. in diameter by 3.5 to 4 cm. in height, with a small extending piece about 7 to 8 cm. in height as a means of removing the cylinder. Cover the beaker and heat to gentle boiling for about 30 min. or until the copper is completely precipitated as a metallic sponge on the aluminum. Thoroughly wash the copper sponge from the aluminum into a 250-ml. beaker and carefully examine the aluminum sheet for any remaining particles of copper. Wash the copper sponge several times by decantation with hot water, filtering the wash water through a 9-cm. filter paper but retaining the copper as completely as possible in the beaker. The filtrate and washings may be discarded or used for the determination of iron, if present (Note 4). Add from 10 to 15 ml. of ferric chloride - hydrochloric acid solution (prepared by dissolving 250 g. of $FeCl_3 \cdot 6H_2O$ in 500 ml. of HCl (1:1)), on the filter paper, catching this in the beaker containing the copper sponge. The copper generally dissolves in a few minutes without the need of heating. Break up any large particles of the copper sponge with a stirring rod. When the copper is completely dissolved, wash the filter thoroughly with cold water and, finally, dilute the filtrate to about 200 ml. with cold water, add 5 ml. of syrupy phosphoric acid, then 3 drops of indicator. Titrate the cold solution at once with 0.1 *N* $K_2Cr_2O_7$ solution (1 ml. of exactly 0.1 *N* $K_2Cr_2O_7$ is equivalent to 0.0032 g. Cu.).

NOTE 3.—A microscopic examination is helpful in showing the presence of metallic Cu, red Cu_2O, and CuO. Washing some of the sample through a fine sieve (No. 325) may reveal the presence of metallic copper retained on the sieve.

NOTE 4.—Commercial cuprous oxides may contain moisture and small amounts of iron, chlorine, oil, and acid insoluble matter.

METALLIC COPPER AND CUPROUS OXIDE[3]

Apparatus

4. *Filter.*[4]—The filter consists of a 55-mm. filter tube of the type used for small Gooch crucibles. In the opening place a perforated porcelain button and seat it at right angles to the stem. Build up an asbestos pad over the button and securely set it by tamping with a glass rod. The filter must be packed in such a way that the tube may be held in an inverted position without dislodging either the disk or the pad. It has been found that a tube about 60 by 25 mm., with a stem 110 mm. in length, is a convenient size. A 20-mm. Gooch crucible button serves to support the asbestos pad.

Special Solutions Required

5. (*a*) *Extraction Solution.*—Dissolve 6 g. of c. p. hydrazine sulfate in 1 liter of NH_4OH (sp. gr. 0.90).

(*b*) *Ferric Chloride Solution.*—Add 150 g. of $FeCl_3 \cdot 6H_2O$ and 300 ml. of HCl (sp. gr. 1.19) to 800 ml. of air-free, carbon dioxide-saturated water.

(*c*) *Indicator.*—Shake 2 g. of barium diphenylamine sulfonate,[5] 50 ml. of carbon dioxide-saturated distilled water, and 5 g. of sodium sulfate until a uniform suspension of barium sulfate is obtained. Dilute the suspension with 50 ml. of water and allow to settle.

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[3] L. C. Hurd and A. R. Clark, "Determination of Metallic Copper in Cuprous Oxide – Cupric Oxide Mixture," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 8, No. 5, p. 380 (1936).

[4] For further information on the filter see W. Geilman, and F. Z. Weibke, *Zeitschrift für anorganische und allgemeine Chemie*, Vol. 199, p. 120 (1931).

[5] Barium diphenylamine sulfonate may be obtained from the Eastman Kodak Co.

When clear, decant the supernatant liquid through a filter and preserve in a dark bottle.

(*d*) *Phosphoric Acid.*—Prepare a solution of 85 per cent reagent ortho-phosphoric acid conforming to specifications of the American Chemical Society.

(*e*) *Potassium Dichromate Solution* (0.1 *N*).—Dissolve 4.903 g. of reagent quality $K_2Cr_2O_7$ in water, and dilute to 1 liter.

(*f*) *Water.*—Boil vigorously distilled water for several minutes and allow to cool in an atmosphere of CO_2. Allow a slow current of CO_2 to pass continuously through the storage bottle.

Procedure for Metallic Copper

6. (*a*) Weigh accurately a sample of suitable size on a small watch glass, and place the glass and contents in a dry, wide-mouth 250-ml. Erlenmeyer flask. If the material is an electrolytic product low in copper, it is advisable to take a 1 to 2-g. sample. If it is from a "thermal process," the percentage of copper is usually large and a smaller sample will suffice. Displace the air in the flask with CO_2 (15 to 20 cu. ft. per hr.) and add 10 ml. of ethyl alcohol to dissolve any oil present in the sample. Without interrupting the flow of CO_2, add 150 ml. of extraction solution (Note 5). Break up any lumps of oxide with a stirring rod by gentle stirring (grinding of the sample with the stirring rod is not necessary). Avoid violent agitation and swishing of the solution. The CO_2 inlet should be about 5 cm. (2 in.) above the surface of the liquid (Note 6). The time required for complete solution of all Cu_2O varies between 1 and 7 min. depending upon the amount and character of the sample under investigation.

(*b*) When the Cu_2O has completely dissolved, as evidenced by the total disappearance of red particles, connect the filter to a suction and slowly lower it into the flask. As soon as the bulk of the solution is removed, rinse the flask with CO_2 saturated water and continue the filtration and washing. Five or six 100-ml. portions of wash water will suffice to remove all the original extraction solution containing the dissolved Cu_2O. It is not necessary that the final wash water remaining in the filter tube be entirely removed. Then disconnect the filter and push the pad and contents back into the flask with a glass rod. Add 15 ml. of $FeCl_3$ solution and warm the flask on a hot plate to just below the boiling point of the solution to dissolve the cupric oxide - copper residue. When all particles have disappeared, cool the solution to below 40 C. and add 10 ml. of phosphoric acid and 3 drops of indicator. Titrate the solution (in an atmosphere of CO_2) by running the $K_2Cr_2O_7$ solution into the flask. The end point is evidenced by a change from pea green to an intense purple color.

(*c*) *Calculation.*—Calculate the percentage of Cu present (Note 7)

$$\text{Cu, per cent} = \frac{A \times B \times 0.03179}{C} \times 100$$

where:

A = normality of $K_2Cr_2O_7$,
B = milliters of $K_2Cr_2O_7$, and
C = weight of sample in grams.

NOTE 5.—If the ammonium hydroxide - hydrazine sulfate solution is allowed to stand in contact with the samples for more than 10 min., the loss in copper may amount to more than 2 mg.

NOTE 6.—Care must be taken not to allow the extraction solution to become saturated with carbon dioxide. The gas inlet to the flask should not be placed beneath the surface of the liquid, to avoid solubility errors.

NOTE 7.—Commercial samples of cuprous oxide have shown from 0.22 to 11.8 per cent Cu.

Procedure for Cuprous Oxide

7. Dissolve 0.2 g. of the sample in ferric chloride and titrate the ferrous

iron produced with $K_2Cr_2O_7$ in the manner described. It is essential that the solution of the sample be carried out in an atmosphere of CO_2 or other inert gas. From the volume of $K_2Cr_2O_7$ solution equivalent to both Cu_2O and Cu, calculate the true percentage of Cu_2O in the sample according to the following formula (Note 8):

$$Cu_2O, \text{ per cent} = (A - B) \times 0.07157 \times N \times 100$$

where:

A = milliters of $K_2Cr_2O_7$ required for Cu plus Cu_2O in 1 g. of sample,

B = milliliters of $K_2Cr_2O_7$ required for Cu in 1 g. of sample = $\frac{\text{percentage of copper}}{N \times 0.03179 \times 100}$, and

N = normality of $K_2Cr_2O_7$.

NOTE 8.—The calculation of the percentage of Cu_2O is simplified if the sample taken for the copper determination be a simple multiple of that in the oxide analysis. In actual practice it has been found that a 1.000-g. sample for metallic copper and a 0.2000-g. sample for cuprous oxide are of convenient size. In this case one-fifth of the volume $K_2Cr_2O_7$ solution used in the determination of metallic copper is subtracted from the total titration of the smaller sample to give the volume of $K_2Cr_2O_7$ solution actually equivalent to the cuprous oxide in the sample.

Report

8. Report the metallic copper (Cu) and the cuprous oxide (Cu_2O). Subtract the copper equivalents from the percentage of total copper as determined in Section 2, calculate the remainder to cupric oxide and report as percentage of CuO (Cu × 1.252 = CuO).

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: K59-1941

Standard Method of

CHEMICAL ANALYSIS OF DRY MERCURIC OXIDE[1]

A.S.T.M. Designation: D 284 – 33

ADOPTED, 1929; REVISED, 1933.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 284; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method covers procedures for the chemical analysis of dry mercuric oxide pigment.

Treatment of Sample

2. If the pigment is lumpy or not finely ground, it shall be ground to a fine powder and thoroughly mixed. Large samples may be thoroughly mixed and a representative portion taken and powdered if lumpy or not finely ground. The sample shall in all cases be thoroughly mixed before taking portions for analysis. All samples shall be preserved in stoppered bottles or containers.

Alkalinity or Acidity

3. Determine the alkalinity or acidity of the sample in accordance with the Standard Method of Test for Alkalinity or Acidity of Pigments (A.S.T.M. Designation: D 278) of the American Society for Testing Materials.[3]

Free Mercury

4. Examine a representative sample of the dry mercuric oxide under a microscope for the presence of free mercury globules.

Combined Mercuric Mercury

5. *Special Solutions Required*: (*a*) *Aqua Regia.*—Mix one volume of HNO_3 (sp. gr. 1.42) and three volumes of HCl (sp. gr. 1.19).

(*b*) *Ammonium Monosulfide.*—Prepare fresh by saturating three volumes of NH_4OH (sp. gr. 0.90) with H_2S and then adding two volumes of NH_4OH (sp. gr. 0.90) to the saturated solution.

(*c*) *Ammonium Polysulfide.*—Dissolve 25 g. of "flowers of sulfur" in a mixture of 500 ml. of ammonium monosulfide and 500 ml. of water.

6. (*a*) *Procedure in the Absence of Other Metals Precipitable as Sulfides.*—Weigh accurately a dry sample of 0.5 g. of the material, transfer it to a 400-ml. beaker, add a solution of 40 ml. of water and 25 ml. of HCl (sp. gr. 1.19), and stir until all soluble matter has dissolved. (Solution should be carried out as near room temperature as possible, since on

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, this method was published as tentative from 1928 to 1929. Editorially revised and rearranged in 1939.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

boiling solutions of mercuric chloride some of the $HgCl_2$ may be volatilized.) Filter off any insoluble matter and wash with warm (not boiling) water until free of chlorides. If appreciable iron is present (otherwise omit this part of the procedure), add sulfurous acid solution in slight excess to the filtrate or original solution to reduce the ferric iron, and warm (do not boil) to expel the excess of sulfur dioxide. Neutralize the solution with NH_4OH, add 1 ml. of HCl (sp. gr. 1.19) and 300 ml. of water. Pass H_2S through the cold solution until saturated, when the precipitate of HgS readily settles leaving a clear supernatant liquid. Filter without delay on a weighed Gooch crucible, wash well (about six times) with cold distilled water and then three times with 10-ml. portions of cold 95 per cent ethyl alcohol. Set the crucible in a small beaker, add sufficient carbon disulfide or carbon tetrachloride to cover the precipitate, cover the beaker with a watch glass, and let stand for about 30 min. Lift the crucible out of the beaker, draining off the solvent, replace in the filtering tube, and wash the precipitate with CCl_4 until 1 ml. of the filtrate leaves no visible residue after evaporation. Wash with three 10-ml. portions of cold 95 per cent ethyl alcohol and once with ethyl ether. Set the crucible on top of an oven until the ether has evaporated, then place it in the oven and dry to constant weight at 105 to 110 C. Cool, and weigh as mercuric sulfide (HgS); calculate the percentage of mercury (Hg) in the sample (HgS × 0.862 = Hg).

(*b*) *Procedure in the Presence of Other Metals Precipitable as Sulfides.*—Treat a dry sample of 0.5 g. of the material as described in Paragraph (*a*) until the sulfides are precipitated, filter on a small paper, and wash with water containing a little H_2S. Transfer the paper and precipitate to a porcelain dish or casserole, spreading the paper out, cover with diluted HNO_3 (sp. gr. 1.2 to 1.3), cover with a watch glass, and boil several minutes. Dilute with water, filter on a small paper, and wash with hot water containing a little HNO_3. Transfer the filter and precipitate to the porcelain dish or casserole, add a little aqua regia, heat gently until the HgS is dissolved and free chlorine expelled, filter off separated sulfur (and $PbSO_4$ if present), wash the residue with hot (not boiling) water until free from chlorides. Nearly neutralize[4] the filtrate with pure Na_2CO_3, add a slight excess of freshly-prepared colorless ammonium monosulfide, and then, with constant agitation, a solution of pure NaOH (free from Ag, Al_2O_3, and SiO_2) until the dark liquid begins to lighten. Heat the solution to boiling and add more NaOH solution until the liquid is perfectly clear. The solution now contains the mercury as a thio-salt (Note). Add an excess of NH_4NO_3 and boil the solution until the ammonia is almost entirely expelled. Let the precipitate settle, and decant the supernatant liquid through a weighed Gooch crucible. Wash the precipitate two or three times by decantation with water containing a little H_2S, then by decantation with hot water until the wash water no longer reacts with $AgNO_3$ solution. Transfer the precipitate to the crucible, wash with 95 per cent ethyl alcohol, treat with CS_2 or CCl_4, and finally weigh the HgS, as described in Paragraph (*a*).

NOTE.—If some $PbSO_4$ should go into solution with the mercury on treating with aqua regia, it will be converted by the ammonium sulfide and NaOH into insoluble PbS, which should be filtered off and washed with diluted NaOH solution.

(*c*) *Procedure in Presence of Arsenic, Antimony or Tin.*—If the material con-

[4] J. Volhard, Liebig's *Ann.*, Vol. 255, p. 255 (1889). Treadwell-Hall, *Analytical Chemistry*, Vol. II, pp. 169, 194, 235 (1914).

tains arsenic, antimony, or tin, treat a dry sample of 0.5 g. as described in Paragraph (*a*) until the sulfides are precipitated, then filter on a small paper and wash with water containing a little H_2S. Transfer the precipitate to a porcelain dish or casserole, cover with ammonium polysulfide, and warm gently for several minutes, stirring constantly. Filter on a small paper and wash with hot water containing ammonium polysulfide. From this point treat the precipitate as described in Paragraph (*b*) for the H_2S precipitate.

Ash

7. Ignite a dry sample of 2 g. of the material in a weighed porcelain crucible or dish under a well-ventilated hood (the fumes are poisonous). Cool and weigh the residue. Calculate the percentage of ash.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: K16.1-1944

Standard Methods of

CHEMICAL ANALYSIS OF DRY RED LEAD[1]

A.S.T.M. Designation: D 49 - 44

ADOPTED, 1918; REVISED, 1927, 1928, 1929, 1933, 1935, 1937, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 49; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods cover procedures for the chemical analysis of dry red lead having the approximate formula Pb_3O_4 (probably $PbO_2 \cdot 2PbO$).

Treatment of Sample

2. If the pigment is lumpy or not finely ground, it shall be ground to a fine powder and thoroughly mixed. Large samples may be thoroughly mixed and a representative portion taken and powdered if lumpy or not finely ground. The sample in all cases shall be thoroughly mixed before taking portions for analysis. All samples shall be preserved in stoppered bottles or containers.

Moisture

3. Dry 2 g. of the sample for 2 hr. at 105 C. The loss in weight is considered as moisture.

Organic Color

4. Boil 2 g. of the sample with 25 ml. of 95 per cent ethyl alcohol, let settle, decant the supernatant liquid; boil the residue with 25 ml. of distilled water and decant as before; boil the residue with 25 ml. of diluted NH_4OH (1:4) and again decant. Boil another 2-g. portion of the sample with 25 ml. of chloroform, let settle, and decant the supernatant liquid. If any one of the above solutions is colored, organic coloring matter is indicated. If the solutions should remain colorless, organic colors are probably absent.

NOTE.—If it is desired to test for organic colors resistant to the above reagents, the test procedures described in the following books may be used, taking into account the nature of the pigment involved: Zerr and Mayer, "Tests for Coal Tar Colors in Aniline Lakes"; Schultz and Julius, "A Systematic Survey of the Organic Coloring Matters"; Mulliken, "Identification of Pure Organic Compounds"; *Commercial Dyestuffs*, Vol. III.

Total Lead and Insoluble Matter

5. (*a*) Treat 1 g. of the sample with 15 ml. of HNO_3 (1:1) and sufficient H_2O_2 to dissolve all PbO_2 on warming. If any insoluble matter is present, add

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these methods were published as tentative from 1917 to 1918.

25 ml. of water, boil, filter, and wash with hot water. The insoluble matter contains free SiO_2 and should be examined for $BaSO_4$ and silicates, if appreciable. To the original solution or filtrate from the insoluble matter add 20 ml. of H_2SO_4 (sp. gr. 1.84) and evaporate to SO_3 fumes. Cool, add 150 ml. of water, and 150 ml. of 95 per cent ethyl alcohol, let stand *cold* for 2 hr., filter on a Gooch crucible, wash with 95 per cent alcohol, dry at 105 to 110 C., and weigh as $PbSO_4$. Calculate to PbO. Red lead is rarely adulterated, but should sample contain soluble barium compounds, the $PbSO_4$ obtained above will contain $BaSO_4$. In this case, precipitate the lead as sulfide from a slightly acid (HCl) solution, dissolve the PbS in hot diluted HNO_3, and determine the lead as sulfate or chromate. If sample contains significant amounts of calcium or magnesium, boil the HNO_3 - H_2O_2 solution until all the lead is converted into nitrate and then determine the lead as $PbCrO_4$.

(*b*) If soluble barium, calcium, or magnesium are to be determined, precipitate the lead as sulfide from a slightly acid solution (HCl), dissolve the PbS in hot diluted HNO_3, and determine the lead as sulfate. Boil the filtrate from the PbS to expel H_2S, add a little bromine water to oxidize iron (if present), boil to expel bromine, and precipitate the barium with a few milliliters of H_2SO_4 (1:3). Filter and weigh as $BaSO_4$. Calculate to BaO or $BaCO_3$. To the filtrate from the $BaSO_4$, add NH_4OH in slight excess, filter off any precipitate of $Fe(OH)_3 + Al(OH)_3$, wash with hot water. Manganese, if present, can be precipitated by adding bromine and NH_4OH and warming. Filter, wash with hot water, ignite, and weigh as Mn_3O_4. Unite all the filtrates, make slightly acid with acetic acid, heat to boiling and pass H_2S into the hot solution until saturated (20 to 30 min.); add 5 g. of NH_4Cl and let stand 5 hr.; filter off any ZnS, wash with H_2S water, dissolve the ZnS in hot diluted HCl and determine the zinc by titration with $K_4Fe(CN)_6$. Or, boil off the H_2S, filter out any separated sulfur and determine the zinc as $Zn_2P_2O_7$. Calcium may be determined in the filtrate from the ZnS by expelling H_2S and then adding NH_4OH and ammonium oxalate. Titrate with $KMnO_4$. In the filtrate from calcium determine magnesium by precipitating with sodium phosphate solution, finally weighing as $Mg_2P_2O_7$.

Lead Peroxide (PbO_2) and True Red Lead (Pb_3O_4)

NOTE.—Method of Diehl[3] modified by Topf[4]—not applicable when substances are present, other than oxides of lead, that liberate iodine under conditions given, or substances such as metallic lead which reduce PbO_2 to PbO without the liberation of iodine.

6. *Solutions Required:* (*a*) *Red Lead Solution.*—Dissolve in a large beaker (about 1.1 liter) 600 g. of reagent grade crystallized sodium acetate and 48 g. of c.p. KI in about 500 ml. of acetic acid solution (25 per cent) (made by mixing 150 ml. of c.p. glacial acetic acid with 450 ml. of distilled water). Warm the beaker and contents on a steam bath, stirring occasionally, until a clear solution is obtained. Cool this solution to room temperature, dilute to exactly 1000 ml. with the acetic acid solution (25 per cent) and mix thoroughly. If preferred, the red lead solution may be prepared separately for each titration, as follows: Dissolve 30 g. of the reagent grade crystallized sodium acetate and 2.4 g. of c.p. KI in 25 ml. of the acetic acid solution (25 per cent), warming gently and stirring until a clear solution is obtained. Cool this solution to room temperature, dilute to 50 ml. with the

[3] *Dingl. polyt. Jour.*, Vol. 246, p. 196.
[4] *Zeitschrift für analytische Chemie*, Vol. 26, p. 296.

acetic acid solution (25 per cent), and mix thoroughly.

(*b*) *Sodium Thiosulfate Solution* (0.1 *N*).—Dissolve 24.83 g. of c.p. sodium thiosulfate ($Na_2S_2O_3 \cdot 5H_2O$), freshly pulverized and dried between filter paper, and dilute with water to 1 liter at the temperature at which the titrations are to be made. The solution is best made with well-boiled water free from CO_2, or let stand 8 to 14 days before standardizing. Standardize with pure, resublimed iodine, as described in Treadwell - Hall, "Analytical Chemistry," Sixth Edition, Vol. II, p. 551 (1924) or in Hillebrand and Lundell, "Applied Inorganic Analysis," p. 154 (1929), and also against pure KIO_3; the two methods of standardization should agree within 0.1 per cent on iodine value.

(*c*) *Starch Solution.*—Stir up 2 to 3 g. of potato starch with 100 ml. of salicylic acid solution (1 per cent), and boil the mixture until the starch is practically dissolved, then dilute to 1 liter (Note), or as per Lord.[5]

NOTE: *Lead Peroxide.*—If sample contains an appreciable amount of nitrite (nitrate has no effect on method), leach out water-soluble matter as below, dry residue and determine PbO_2 as above, calculating to basis of original sample.

7. *Procedure.*—Weigh 1 g. of the finely-ground sample, transfer to a 200-ml. Erlenmeyer flask, add 20 ml. of distilled water; then add as quickly as possible 40 ml. of the "red lead solution" at room temperature. If the sample is red lead which has been extracted from the paint or paste, in place of the distilled water use 10 ml. of a mixture of 7 parts by volume of chloroform and 3 parts by volume of c.p. glacial acetic acid, and then add without delay the red lead solution. Add 30 ml. of water containing 5 or 6 g. of sodium acetate and titrate at once with $Na_2S_2O_3$ (0.1 *N*), adding the latter rather slowly and keeping the liquid constantly in motion by whirling the flask. When the solution has become light yellow, rub-up any undissolved particles with the rod until free iodine no longer forms, wash off rod, add the sodium thiosulfate solution until *pale yellow*, add starch solution, and titrate until colorless. Add the iodine solution (0.1 *N*) until blue color is just restored and subtract the amount used from the volume of sodium thiosulfate that had been added.

8. *Calculation.*—Calculate the lead peroxide and true red lead contents as follows:

$$PbO_2 = S \times 0.942$$
$$Pb_3O_4 = PbO_2 \times 2.86616 = S \times 2.7$$

where:

S = iodine value of the sodium thiosulfate solution.

Zinc

9. If in appreciable amount, evaporate off the alcohol from the filtrate from total lead, make alkaline with NH_4OH, then acid with HCl (sp. gr. 1.19), add 3 ml. more of HCl, dilute to about 250 ml. with water, heat nearly to boiling and titrate with standard $K_4Fe(CN)_6$ solution as described by Low.[6] Report as ZnO (includes cadmium). Iron, copper, or other interfering substances should first be removed as described by Low.

Matter Soluble in Water

10. Transfer 25 g. of the pigment to a graduated 250-ml. flask, add 100 ml. of water, and boil for 5 min. Cool to room temperature, dilute to the mark with water, mix, and allow to settle. Filter the supernatant liquid through a dry filter paper and discard the first 20 ml. of the filtrate. Transfer 100 ml. of the

[5] "Notes on Metallurgical Analysis," p. 103 (1903).

[6] Low, "Technical Methods of Ore Analysis."

clear filtrate to a weighed dish, evaporate to dryness on a steam bath, dry for 1 hr. in an oven at 105 to 110 C., cool, and weigh. Calculate the percentage of water-soluble matter.

Total Silica

11. Digest 5 g. of the sample in a covered casserole with 5 ml. of HCl and 15 ml. of HNO_3 (1:1). Evaporate to dryness to dehydrate. Cool, treat with hot water and HNO_3, boil, filter, wash with hot acid ammonium acetate solution, then diluted HCl and finally hot water. Ignite and weigh as SiO_2. The residue may be treated with H_2SO_4 and HF in cases of doubt as to purity.

Carbon Dioxide

12. Determine carbon dioxide by the evolution method, using diluted HCl and stannous chloride.

Soluble Sulfates

13. (*a*) *Sulfates other than Barium Sulfate.*—Treat 0.5 g. of the sample with 5 ml. of water, 3 g. of NH_4Cl, and 5 ml. of HCl saturated with bromine. Digest (covered) on steam bath about 15 min. Add 25 ml. of water, neutralize with dry Na_2CO_3, and add about 2 g. more. Boil 10 to 15 min., let settle, dilute with hot water, filter, and wash with hot water. Redissolve in HCl, reprecipitate as above, and wash thoroughly with hot water. Acidify united filtrates with HCl, adding a slight excess; boil and add slight excess of $BaCl_2$ solution (10 per cent). Let stand on steam bath for 1 hr. Filter, and wash with hot water. Ignite, and weigh as $BaSO_4$. Calculate to SO_3 (includes SO_3 formed from SO_2).

(*b*) Or, dissolve 0.5 g. of the sample in 25 ml. of water, 10 ml. of NH_4OH (sp. gr. 0.90) and HCl in slight excess; dilute to about 150 ml. with water and add a piece of aluminum foil which should about cover the bottom of the beaker (being held on the bottom by means of a stirring rod). Heat gently till all lead is precipitated, decant through a filter, pressing the lead sponge with a flattened rod, and washing with hot water. Add to the filtrate a little pure bromine water, boil until bromine is expelled, add 15 ml. of $BaCl_2$ solution (10 per cent), let stand on steam bath for 1 hr., filter, wash with hot water, ignite, and weigh as $BaSO_4$ (any $SrSO_4$ present is not decomposed in this method).[7]

Iron Oxide

14. Determine iron oxide by Schaeffer's[8] modification of Thomson's colorimetric method; or, in a large beaker, treat 20 g. of the sample with 20 ml. of water, 20 ml. of HNO_3 (sp. gr. 1.42), and 3 ml. of formaldehyde solution. Warm until all PbO_2 is dissolved, dilute with water, warm, filter off insoluble matter, and wash with hot water. Ignite filter and insoluble matter, and evaporate with H_2SO_4 and HF. To filtrate from insoluble matter add 14 ml. of H_2SO_4 (1:1), filter off $PbSO_4$, and wash. Dissolve residue from HF and H_2SO_4 in H_2SO_4 and add to filtrate from $PbSO_4$. Dilute to 500 ml. and determine iron colorimetrically in an aliquot, using the same amounts of HNO_3, H_2SO_4, and formaldehyde in comparison solution.[9] Calculate to Fe_2O_3.

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[7] The solubility of $BaSO_4$ is increased by the presence of aluminum chloride. See J. W. Mellor, "A Treatise on Quantitative Inorganic Analysis," p. 615, J. B. Lippincott Co., Philadelphia, Pa. (1913).

[8] *Journal of Industrial and Engineering Chemistry*, Vol. 4, p. 659 (1912).

[9] Lunge-Berl, "Chemisch-technische Untersuchungs-Methoden," Vol. 2, p. 95, 6th Ed.

APPROVED AS
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Standard Methods of

CHEMICAL ANALYSIS OF WHITE PIGMENTS[1]

A.S.T.M. Designation: D 34 – 39

ADOPTED, 1917; REVISED, 1927, 1930, 1933, 1939.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 34; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods cover procedures for the chemical analysis of white pigments. The procedures appear in the following order:

	Section
Preparation of Sample	2
Small Amounts of Iron	3
Lead Pigments:	
Basic carbonate of lead	4 to 9
Basic sulfate of lead	10 to 16
Zinc Pigments:	
Zinc-lead and leaded-zincs (ozlo white)	17 to 20
Zinc oxide	21 to 24
Lithopone	25 to 28
Titanium Pigments	29 to 38
Calcium Pigments:	
Whiting, Paris white, Spanish white, Chalk	39 to 41
Gypsum, Terra alba, Plaster of Paris	42 and 43
Barium Pigments:	
Barytes or barite, "blanc fixe"	44 to 46
Silica Pigments:	
Silica or silex	47
China clay	48
Asbestine	49

Preparation of Sample

2. Dry pigments, if lumpy or not finely ground, shall be ground to a fine powder for analysis. Large samples may be thoroughly mixed and a representative portion taken and powdered if lumpy or not finely ground. The sample in all cases shall be thoroughly mixed before taking portions for analysis. Extracted pigments shall be ground to a fine powder, passed through a No. 80 (177-micron) sieve[3] to remove any skins, and thoroughly mixed. The weighed portions of extracted pigments should be moistened with a little alcohol before adding reagents for analysis. All samples shall be preserved in stoppered bottles or containers.

Small Amounts of Iron

3. (*a*) *Lead Pigments.*—In the case of lead pigments, treat the sample with HNO_3 (1:1), dilute with water, add H_2SO_4 to precipitate bulk of lead (not necessary to evaporate down). Cool,

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these methods were published as tentative from 1915 to 1917, being revised in 1917. In 1939 Standard Methods D 186 were incorporated in this standard.

[3] Detailed requirements for this sieve are given in the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

filter, wash with diluted H_2SO_4 (1:99), make filtrate just alkaline with NH_4OH, then just acid with diluted HNO_3; determine iron colorimetrically by the thiocyanate method,[4] using the same amounts of reagents in preparing the reference color standards. If the sample contains insoluble matter, filter out and wash with hot water until lead-free, and to the filtrate add H_2SO_4 and proceed as given. Ignite the insoluble matter, treat with HF and H_2SO_4, bring into solution (filter out any $BaSO_4$), and add to filtrate from $PbSO_4$. If necessary, the solution may be diluted to volume and aliquots taken. Copper, if present, shall be removed by precipitating the Fe with NH_4OH, filtering, washing, redissolving $Fe(OH)_3$, and proceeding as above.

(*b*) *Other Pigments.*—In the case of pigments other than lead pigments, treat as described in Paragraph (*a*) omitting the addition of H_2SO_4.

LEAD PIGMENTS

Basic Carbonate of Lead

Special Solution Required

4. *Standard Ammonium Molybdate.*—Dissolve 4.25 g. of ammonium molybdate in water and dilute to 1 liter. To standardize this solution, dissolve about 0.2 g. of pure lead foil (pure PbO or $PbSO_4$ may also be used) in HNO_3, evaporate nearly to dryness, add 30 ml. of water, then 5 ml. of H_2SO_4 (sp. gr. 1.84), cool, and filter. Drop paper with $PbSO_4$ into a flask, add 10 ml. of HCl (sp. gr. 1.19), boil until completely disintegrated, add 15 ml. of HCl (sp. gr. 1.19), 25 ml. of water, and NH_4OH until alkaline. Acidify with acetic acid, dilute to 200 ml. with hot water, and boil. Titrate with the molybdate solution, using an outside indicator of one part of tannic acid in 300 parts of water.

[4] Described in Scott "Standard Methods of Analysis," Second Edition, p. 222, D. Van Nostrand Co.

Total Lead, Gravimetric

5. (*a*) *As Sulfate.*—Dissolve 1 g. of the sample in 20 ml. of HNO_3 (1:1) in a covered beaker, heating until all CO_2 is expelled. Wash off cover, add 20 ml. of H_2SO_4 (1:1), and evaporate to fumes of H_2SO_4. Cool, add about 150 ml. of water and 150 ml. of ethyl alcohol. Let stand in *cold* water 1 hr., filter on a Gooch crucible, wash with 95 per cent ethyl alcohol, dry at 110 C., and weigh $PbSO_4$. Calculate to PbO or to basic carbonate ($2PbCO_3 \cdot Pb(OH)_2$).[5]

(*b*) *As Dichromate.*—Dissolve 1 g. of the sample by boiling with acetic acid. Dilute to about 200 ml. with water, make alkaline with NH_4OH, then acid with acetic acid, heat to boiling, and add 10 to 15 ml. of $K_2Cr_2O_7$ (10 per cent). Heat until the yellow precipitate assumes an orange color. Let settle, and filter on a Gooch crucible, washing by decantation with hot water until the washings are colorless, finally transferring all of the precipitate. Wash with 95 per cent ethyl alcohol and then ether, dry at 110 C., and weigh $PbCrO_4$. (Any insoluble matter should be filtered out before precipitating the lead.)

Total Lead, Volumetric

6. (*a*) Dissolve 0.5 g. of the sample in 10 ml. of HCl (sp. gr. 1.19), boil until solution is effected, cool, dilute to 40 ml., neutralize with NH_4OH. Add acetic acid until distinctly acid, dilute to 200 ml. with hot water, boil, and titrate with the standard ammonium molybdate, using an outside indicator of one part of tannic acid in 300 parts of water.

(*b*) It should be noted that when calcium is present, it forms a more or less insoluble molybdate, and results are likely to be high. With samples containing less than 10 per cent of lead, the lead should be precipitated as $PbSO_4$,

[5] This method of weighing lead sulfate is not accurate in the presence of calcium compounds.

filtered, redissolved, and titrated as in the process of standardizing the molybdate solution (Section 4).

Carbon Dioxide

7. Determine by evolution with diluted HCl, absorbing in soda lime or KOH solution. Calculate CO_2 to $PbCO_3$, subtract PbO equivalent from total PbO, and calculate residual PbO to $Pb(OH)_2$.

Acetic Acid[6]

8. Place 18 g. of the sample in a 500-ml. flask; add 40 ml. of sirupy phosphoric acid (H_3PO_4), 18 g. of zinc dust, and 50 ml. of water. Connect to a straight Liebig condenser, apply heat, and distill down to a small bulk. Then pass steam into the flask until it becomes about half full of condensed water, shut off the steam, and distill down to a small bulk—this operation being conducted twice. To the total distillate which was collected in a larger flask, add 1 ml. of H_3PO_4, connect to a Liebig condenser, using a spray trap, and distill to a small volume (about 20 ml). Pass steam through until about 200 ml. of water condense in the distillation flask, shut off the steam, and continue the distillation. Continue these operations of direct and steam distillations until 10 ml. of the distillate require only one drop of 0.1 *N* alkali to give a change in the presence of phenolphthalein. Then titrate the total distillate with 0.1 *N* NaOH, using phenolphthalein as indicator, and calculate the total acidity as acetic acid. It will be found convenient to titrate each 200-ml. portion of the distillate as collected.

Lead

9. (*a*) *As Metallic Lead.*—Weigh 50 g. of the sample into a 400-ml. beaker, add a little water, and add slowly 60 ml. of 40 per cent acetic acid. After effervescence has ceased, boil on hot plate. Fill the beaker with water, let settle, and decant the clear solution. To the residue, add 100 ml. of a mixture of 360 ml. of NH_4OH (sp. gr. 1.19), 1080 ml. of water, 2160 ml. of 80 per cent acetic acid and boil until all solution is complete. Fill beaker with water, let settle, and decant the clear solution. Collect the residue on a watch glass, floating off everything but metallic lead. Dry and weigh. The result multiplied by two is equivalent to the percentage of metallic lead in the sample.

(*b*) *Lead Hydroxide.*—The following method of A. N. Finn (unpublished) gives total basicity of a pure white lead: Place 2 g. of the sample in an evolution flask, add a little CO_2-free water, connect up to the separatory funnel and condenser (Knorr type), add through the funnel, finally washing down, 100 ml. of 0.25 *N* HNO_3. Boil, and absorb the CO_2 in a soda-lime tube (having H_2SO_4 and $CaCl_2$ drying tubes in train) and weigh. To the solution in the evolution flask add about 20 ml. of neutral Na_2SO_4 solution and titrate with 0.25 *N* NaOH (carbonate-free), using phenolphthalein as indicator. Calculate CO_2 to $PbCO_3$. Calculate the amount of 0.25 *N* acid corresponding to the CO_2, and deduct from the total amount of 0.25 *N* acid neutralized by the sample. Calculate the difference to combined water and from this calculate $Pb(OH)_2$.

Basic Sulfate of Lead

Special Solution Required

10. *Standard Potassium Ferrocyanide Solution.*—Dissolve 22 g. of pure $K_4Fe(CN)_6 \cdot 3H_2O$ in water, and dilute to 1000 ml. To standardize, transfer about 0.2 g. (accurately weighed) of pure metallic zinc or freshly ignited pure ZnO to a 400-ml. beaker. Dissolve

[6] Thompson's Method, *Journal*, Soc. Chemical Industry, Vol. 24, p. 487 (1905).

in 10 ml. of HCl and 20 ml. of water. Drop in a small piece of litmus paper, add NH_4OH until slightly alkaline, then add HCl until just acid, and finally add 3 ml. of HCl (sp. gr. 1.19). Dilute to about 250 ml. with hot water and heat nearly to boiling. Run in $K_4Fe(CN)_6$ slowly from a burette, while stirring constantly, until a drop tested on a white porcelain plate with a drop of the uranyl indicator (a 5 per cent solution of uranyl nitrate in water or a 5 per cent solution of uranyl acetate in water made slightly acid with acetic acid) shows a brown tinge after standing 1 min. Run a blank with the same amounts of reagents and water as in the standardization. Subtract the amount of $K_4Fe(CN)_6$ required for the blank from the amounts used in standardization and in titration of the sample (Section 14).

Qualitative Analysis

11. By the usual methods of qualitative analysis, test the sample for matter insoluble in acid ammonium acetate solution, for calcium, for carbonates, and for any other impurities suspected.

Moisture

12. Place 1 g. of the sample in a tared, wide-mouth, short, weighing tube provided with a glass stopper. Heat with stopper removed for 2 hr. at 105 to 110 C. Insert stopper, cool, and weigh. Calculate loss in weight as moisture.

Insoluble Matter and Total Lead

13. In a 250-ml. beaker, moisten 1 g. of the sample with a few drops of alcohol; add 50 ml. of acid ammonium acetate solution (made by mixing 150 ml. of 80 per cent acetic acid, 100 ml. of water, and 95 ml. of NH_4OH (sp. gr. 0.90)). Heat to boiling and boil for 2 min. Decant through a filter paper, leaving any undecomposed matter in the beaker. To the residue in the beaker, add 50 ml. of the acid ammonium acetate solution, heat to boiling, and boil for 2 min. Filter through the same paper and wash with hot water. If an appreciable residue remains, ignite and weigh as insoluble matter. Unite the acid ammonium acetate solutions, heat to boiling, and add drop by drop, while stirring, a slight excess (in total about 10 to 15 ml.) of a solution of $K_2Cr_2O_7$ (10 per cent). Heat until the precipitate assumes an orange color, let settle, filter on a weighed Gooch crucible, wash by decantation with hot water until the washings are colorless, and finally transfer all of the precipitate to the crucible. Then wash with 10 ml. of 95 per cent ethyl alcohol and finally with 10 ml. of ethyl ether. Dry at 105 to 110 C., cool, and weigh $PbCrO_4$. Calculate to PbO by multiplying by the factor 0.69.

Zinc Oxide

14. Weigh accurately about 1 g. of the sample, transfer to a 400-ml. beaker, add 30 ml. of HCl (1:2), boil for 2 or 3 min., add 200 ml. of water and a small piece of litmus paper, add NH_4OH until slightly alkaline, render just acid with HCl, then add 3 ml. of HCl (sp. gr. 1.19), heat nearly to boiling, and titrate with standard potassium ferrocyanide as in standardizing that solution (Section 10). The titration must be made under the same conditions of temperature, volume, and acidity as obtained when the standardization was made. Calculate total zinc as ZnO.

Total Lead Sulfate

15. In a 400-ml. beaker, moisten 0.5 g. of the sample with a few drops of alcohol; add 10 ml. of bromine water, 10 ml. of HCl (1:1), and 3 g. of NH_4Cl. Cover with a watch glass and heat on a steam bath for 5 min., add hot water to give a total volume of about 200 ml., boil for

5 min., filter to separate any insoluble matter (a pure pigment should be completely dissolved), and wash thoroughly with hot water. (The insoluble matter may be ignited, weighed, and examined qualitatively.) Neutralize the clear solution (original solution or filtrate from insoluble matter) in a covered beaker with dry Na_2CO_3, add 1 g. more of dry Na_2CO_3, and boil for 10 to 15 min. Wash off cover, let settle, filter, and wash with hot water. Redissolve the precipitate in HCl (1:1), reprecipitate with Na_2CO_3 as above, filter, and wash thoroughly with hot water. Acidify the united filtrates with HCl, adding about 1 ml. in excess. Boil to expel bromine, and to the clear boiling solution add slowly, while stirring, 15 ml. of $BaCl_2$ (10 per cent). Let stand on a steam bath for about 1 hr., filter on a weighed Gooch crucible, wash thoroughly with boiling water, dry, ignite, cool, and weigh as $BaSO_4$. Calculate to total $PbSO_4$, using the factor 1.3.

Calculations and Report

16. Calculate the percentage of total $PbSO_4$ to PbO by multiplying by the factor 0.736 and subtract the result from the percentage of PbO found under insoluble impurity and total lead in Section 13. Report the difference as PbO. Report ZnO found under zinc oxide in Section 14, as percentage of ZnO. Report moisture and insoluble matter as such.

ZINC PIGMENTS

Zinc-Lead and Leaded-Zincs (Ozlo White)

Total Lead and Zinc

17. Dissolve 1 g. of the sample by boiling with 250 ml. of water and 20 ml. of HNO_3 (sp. gr. 1.42), add 5 ml. of H_2SO_4 (sp. gr. 1.84), and evaporate to copious fumes of H_2SO_4. Cool, add 250 ml. of water, let stand *cold* 1 to 2 hr., filter on Gooch crucible, wash with H_2SO_4 (1:99), ignite, and weigh as $PbSO_4$. Report as $PbSO_4$. The $PbSO_4$ may be filtered on paper and then dissolved and titrated with ammonium-molybdate solution as described in Section 4. Make filtrate from $PbSO_4$ alkaline with NH_4OH, then acid with HCl, add 3 ml. of HCl (sp. gr. 1.19), heat nearly to boiling, and titrate the total zinc with $K_4Fe(CN)_6$ solution as described in Section 10, using uranium-acetate solution as an outside indicator. Calculate to ZnO. (Iron, copper, or other interfering substances should first be removed as described by Low.[7])

NOTE.—If sample contains Ca or Mg, the Pb and Zn should be separated by precipitation with H_2S after dissolving in HCl, making alkaline with NH_4OH and then acid with acetic acid. The PbS + ZnS is dissolved in diluted HNO_3 and the Pb and Zn determined as above.

Sulfates other than Barium Sulfate

18. (*a*) *Method A.*—Treat 0.5 g. of the sample with 5 ml. of water, 3 g. of NH_4Cl, and 5 ml. of HCl saturated with bromine. Digest (covered) on steam bath about 15 min., add 25 ml. of water, neutralize with dry Na_2CO_3 and add about 2 g. more, boil 10 to 15 min. Let settle, dilute with hot water, filter, and wash with hot water. Redissolve in HCl, reprecipitate as above, and wash thoroughly with hot water. Acidify united filtrates with HCl, adding a slight excess. Boil and add slight excess of $BaCl_2$ (10 per cent). Let stand on steam bath 1 hr., filter, wash with hot water, ignite, and weigh $BaSO_4$. Calculate to SO_3 (includes SO_3 formed from SO_2).

(*b*) *Method B.*—Dissolve 0.5 g. of the sample in 25 ml. of water, 10 ml. of NH_4OH (sp. gr. 0.90), and HCl in slight excess. Dilute to about 150 ml. with water and add a piece of aluminum foil

[7] Low, "Technical Methods of Ore Analysis," 7th Edition, p. 288, John Wiley and Sons, New York City (1914).

which should about cover the bottom of the beaker, the foil being held on the bottom by means of a stirring rod. Heat gently until all lead is precipitated, decant through a filter, pressing the lead sponge with a flattened rod, and washing with hot water. Add to the filtrate a little pure bromine water, boil until bromine is expelled, add 15 ml. of $BaCl_2$ (10 per cent), let stand on steam bath 1 hr., filter, wash with hot water, ignite, and weigh as $BaSO_4$ (any $SrSO_4$ present is not decomposed in this method).[8]

Soluble Zinc Sulfate

19. To 2 g. of the sample add 150 ml. of water and 50 ml. of 95 per cent alcohol and heat nearly to boiling for 30 min., filter, and wash with a mixture of alcohol and water (1:3). Heat filtrate to boiling and expel most of the alcohol; then determine SO_3 by usual method of precipitation with $BaCl_2$. Calculate to $ZnSO_4$ and to SO_3.

Sulfur Dioxide

20. Determine as described in Section 23 under the analysis of zinc oxide.

Zinc Oxide

Total Zinc

21. Dissolve 0.25 to 0.3 g. of the sample in 10 ml. of HCl (sp. gr. 1.19) and 20 ml. of water, make alkaline with NH_4OH, then acid with HCl, add 3 ml. more of HCl (sp. gr. 1.19), dilute to about 250 ml. with water, heat nearly to boiling, and titrate with standard $K_4Fe(CN)_6$ solution as described in Section 10. Report as ZnO (includes Cd). Iron, copper, or other interfering substances should be first removed as described by Low.[9]

[8] The solubility of $BaSO_4$ is increased by the presence of aluminum chloride. See J. W. Mellor, "A Treatise on Quantitative Inorganic Analysis," p. 615, J. B. Lippincott Co., Philadelphia, Pa. (1913).

[9] Low, "Technical Methods of Ore Analysis," 7th Edition, p. 288, John Wiley and Sons, New York City (1914).

Total Soluble Sulfur[10]

22. Moisten 10 g. of the sample with water, add a few drops of bromine and then HCl (sp. gr. 1.19), boil to expel bromine, filter from any insoluble matter, and wash with hot water. Make alkaline with NH_4OH, then just slightly acid with HCl, heat to boiling, and add about 15 ml. of hot $BaCl_2$ solution. Let stand several hours (overnight), filter on a weighed Gooch crucible, wash well with hot water, dry, ignite for 5 min., cool and weigh as $BaSO_4$. Calculate to S.

Sulfur Dioxide[10]

23. Mix 5 g. of the sample with 50 ml. of warm (freshly boiled and then partly cooled) water to an emulsion and pour into a glass-stoppered flask containing 18 ml. of HCl (sp. gr. 1.19) and exactly 25 ml. of 0.1 *N* iodine solution, stopper, and shake until all the oxide is dissolved. Titrate the excess of iodine as rapidly as possible with 0.1 *N* sodium thiosulfate solution. Calculate to SO_2.

Soluble Zinc Sulfate

24. Determine as described in Section 19 under the analysis of zinc-lead and leaded-zincs.

Lithopone

Special Solutions Required

25. (*a*) *Alkaline Lead Nitrate Solution.*—Into 100 ml. of KOH solution (56 g. in 140 ml. of water) pour a saturated solution of $Pb(NO_3)_2$ (250 g. in 500 ml. of water) until the precipitate ceases to redissolve, stirring constantly while mixing. About three volumes of the $Pb(NO_3)_2$ solution will be required for one volume of the KOH solution.

(*b*) *Cadmium Chloride Solution.*—Dissolve 8 g. of $CdCl_2$ in 250 ml. of water; add 150 ml. of NH_4OH (sp. gr. 0.90).

[10] Method of G. Rigg.

(*c*) *Standard Iodine Solution.*—Dissolve about 12.7 g. (accurately weighed) of pure resublimed iodine and 18 g. of KI in a small volume of water and then dilute to 1 liter.

Insoluble Matter and Total Zinc

26. To 1 g. of the sample in a 200-ml. beaker, add 10 ml. of HCl (sp. gr. 1.19), mix, and add in small portions about 1 g. of $KClO_3$, then heat on the steam bath until about half of the liquid is evaporated. Dilute with water, add 5 ml. of diluted H_2SO_4 (1:10). Boil, let settle, filter, wash, ignite, cool, and weigh the insoluble matter which should be only $BaSO_4$. Make a qualitative examination for aluminum oxide and silicon dioxide. Examine the insoluble matter under the microscope for the presence of natural crystalline barytes or the sample may also be examined direct. Make the filtrate from the insoluble matter alkaline with NH_4OH, acid with HCl, add 3 ml. of HCl (sp. gr. 1.19), dilute to about 250 ml. Heat nearly to boiling, and titrate with the standard $K_4Fe(CN)_6$ solution as described in Section 10. Calculate to Zn.

Zinc Oxide

27. Weigh accurately 1 g. of the sample, transfer to a 250-ml. beaker (moisten with a few drops of alcohol if an extracted pigment), add about 100 ml. of acetic acid (3:97), stir vigorously but do not heat, cover, and let stand for 18 hr., stirring once every 5 min. for the first 30 min. Filter, wash with acetic acid (3:97) followed by water until the washings give no test for zinc with $K_4Fe(CN)_6$ solution. Dilute the clear filtrate to about 200 ml. with water, add 30 ml. of HCl (1:2) and a small piece of litmus paper, add NH_4OH (sp. gr. 0.90) until slightly alkaline, render just acid with HCl, then add 3 ml. of HCl (sp. gr. 1.19). Heat nearly to boiling, and titrate with standard $K_4Fe(CN)_6$ solution as described in Section 10. Calculate to ZnO. Calculate this result to Zn, subtract from total Zn, and calculate the difference to ZnS. (Any $ZnCO_3$ or $ZnSO_4$ is included in the ZnO.)

Zinc Sulfide[11]

28. (*a*) Place 0.5 g. of the sample in an evolution flask with about 10 g. of feathered or mossy zinc, add 50 ml. of water, and insert the stopper carrying a separatory funnel and an exit tube. Run in 50 ml. of HCl (sp. gr. 1.19) from the funnel, having previously connected the exit tube to two absorption flasks in series (first flask containing 100 ml. of alkaline lead nitrate solution (Section 25), second flask, 50 ml. of same as a safety device). After all of the acid has run into the evolution flask, heat slowly, finally boiling until the first appearance of steam in the first absorption flask. Disconnect, let the PbS settle, filter; wash with cold water, then with hot water till neutral to litmus paper and washings give no test for lead. Dissolve the PbS precipitate in hot, diluted HNO_3, evaporate to fumes with H_2SO_4 and finally weigh as $PbSO_4$. Calculate PbS or $PbSO_4$ to ZnS.

(*b*) Instead of absorbing the evolved H_2S in alkaline lead nitrate solution, cadmium chloride solution (Section 25), may be used. The CdS precipitate may be filtered on a weighed Gooch crucible, washed with water containing a little NH_4OH, dried at 100 C., and weighed. Calculate to ZnS. It is better to filter the CdS on a small filter and wash as above; then place filter and precipitate in a beaker and dissolve in HCl and $KClO_3$ (keeping at room temperature at first), filter out any paper pulp or insoluble matter. Make filtrate alkaline

[11] Evolution Method of W. G. Scott, "White Paints and Painting Material," The Modern Painter, p. 257 (1910); see also Blair, "Chemical Analysis of Iron," 8th Edition, p. 55, J. B. Lippincott Co., Philadelphia, Pa. (1918).

with NH_4OH, then just acid with HCl, heat to boiling, and precipitate with $BaCl_2$. Filter, wash, ignite, and weigh $BaSO_4$. Calculate to ZnS.

(*c*) For very rapid work the contents of the absorption flask, after all H_2S has been absorbed, may be washed into a vessel with *cold* water and diluted to about 1 liter, acidified with HCl (sp. gr. 1.19), and titrated with standard iodine solution (Section 25), using starch indicator. (The precipitate should be completely dissolved.)

TITANIUM PIGMENTS

Special Solutions Required

29. (*a*) *Standard Potassium Permanganate Solution (0.1 N).*—Dissolve 3.2 g. of pure $KMnO_4$ in 1 liter of distilled water, let stand 8 to 14 days, siphon off the clear solution (or filter through purified asbestos), and standardize against the National Bureau of Standards standard sample No. 40 of sodium oxalate as follows: In a 400-ml. beaker dissolve 0.25 to 0.30 g. (accurately weighed) of the Bureau of Standards sodium oxalate in 250 ml. of hot water (80 to 90 C.) and add 15 ml. of diluted H_2SO_4 (1:1). Titrate at once with the $KMnO_4$ solution, *stirring the liquid vigorously and continuously.* The $KMnO_4$ must not be added more rapidly than 10 to 15 ml. per min., and the last 0.5 to 1 ml. must be added drop by drop with particular care to allow each drop to be fully decolorized before the next is introduced. The solution should not be below 60 C. by the time the end point is reached. (More rapid cooling may be prevented by allowing the beaker to stand on a small asbestos-covered hot plate during the titration. The use of a small thermometer as a stirring rod is most convenient.) The weight of $Na_2C_2O_4$ used multiplied by 0.833 gives its iron equivalent, or multiplied by 1.195 gives its titanium dioxide equivalent. The $KMnO_4$ solution should be kept in a glass-stoppered bottle painted black to keep out light.

(*b*) *Ferric Sulfate Solution for Titanium, (2 per cent Fe).*—Dissolve 20 g. of pure iron or plain carbon steel in a slight excess of HCl (sp. gr. 1.19), oxidize with HNO_3 (sp. gr. 1.42), add about 80 ml. of H_2SO_4 (sp. gr. 1.84), and heat until fumes of the latter are evolved. Cool, dilute with water to 1000 ml., digest on a steam bath until sulfates are dissolved, and filter if necessary. Add 0.1 *N* $KMnO_4$ solution until a faint pink color persists for 5 min. (to oxidize any ferrous iron that may be present).

Ferric ammonium sulfate may be used also.[12]

(*c*) *Standard Ferric Sulfate Solution for Colorimetric Determination of Iron.*—Determine the strength of the ferric solution for the TiO_2 determination in terms of Fe and dilute a portion of this solution until one is obtained of such strength that 1 ml. is equivalent to 0.00001 g. of Fe.

(*d*) *Thiocyanate Indicator.*—Prepare a 2 per cent solution of pure KCNS or NH_4CNS in distilled water.

Color

30. To 5 g. of the sample add 1.5 ml. of linseed oil, rub up on a stone slab or glass plate with a flat-bottom glass or stone pestle or muller to a uniform smooth paste. Treat in a similar manner 5 g. of the reference standard titanium pigment. Spread the two pastes side by side on a clear, colorless glass plate and compare the colors. If the sample is as white as, or whiter than the reference standard, it passes this test.

Qualitative Analysis

31. Place about 0.5 g. of the sample in a 250-ml. resistant-glass beaker,[13] add

[12] Gooch, "Methods in Chemical Analysis," 1st Edition, p. 436.

[13] Pyrex glass is very satisfactory for this purpose.

20 ml. of H_2SO_4 (sp. gr. 1.84) and 7 to 8 g. of $(NH_4)_2SO_4$. Mix well and boil for a few minutes. The sample should go completely into solution; a residue denotes the presence of SiO_2 or siliceous matter. Cool the solution, dilute with 100 ml. of water, heat to boiling, let settle, filter, wash with hot diluted H_2SO_4 (1:19) until free from titanium, and test the residue for lead, etc. Test the filtrate for calcium, zinc, iron, chromium, etc., by the regular methods of qualitative analysis. For the iron determination add to a portion of the filtrate 5 g. of tartaric acid, render slightly ammoniacal, pass in H_2S in excess, and digest at side of steam bath for a while. No precipitate indicates the absence of iron, nickel, cobalt, lead, copper, etc. A black precipitate readily soluble in diluted HCl denotes iron. For titanium, test a small portion of the original filtrate with H_2O_2 (a clear yellow-orange color should result) and another portion with metallic tin or zinc (a pale blue to violet coloration should result). Negative tests should be shown for sulfur present as sulfide, carbonates, and appreciable water-soluble matter.

Moisture

32. Place 1 g. of the sample in a wide-mouthed, short weighing tube provided with a glass stopper. Heat, with stopper removed, for 2 hr. at 105 to 110 C. Insert stopper, cool, and weigh. Report the loss in weight as moisture.

Matter Soluble in Water (In the case of Titanium-Barium Pigments)

33. Transfer 2.5 g. of the sample to a graduated 250-ml. flask, add 100 ml. of water, and boil for 5 min. Cool to room temperature, dilute to the mark with water, mix, and allow to settle. Filter the supernatant liquid through a dry filter paper and discard the first 20 ml. of the filtrate. Transfer 100 ml. of the clear filtrate to a weighed dish and evaporate to dryness on a steam bath. Dry for 1 hr. in an oven at 105 to 110 C., cool, and weigh. Calculate the percentage of water-soluble matter.

Titanium Oxide

34. Transfer 0.5 g. of the dried sample to a 250-ml. resistant-glass beaker,[13] add 20 ml. of H_2SO_4 (sp. gr. 1.84) and 7 to 8 g. of $(NH_4)_2SO_4$. Mix well and heat on hot plate until fumes of H_2SO_4 are evolved, and then continue the heating over a strong flame until solution is complete (usually requires not over 5 min. of boiling) or it is apparent that the residue is composed of SiO_2 or siliceous matter. Caution should be observed in visually examining this hot solution. Cool the solution, dilute with 100 ml. of water, stir, heat carefully to boiling while stirring, let settle, filter through paper and transfer the precipitate completely to the paper. Wash the insoluble residue with cold diluted H_2SO_4 (1:19) until titanium is removed. Dilute the filtrate to 200 ml. and add about 5 ml. of NH_4OH (sp. gr. 0.90) to lower the acidity to approximately 10 to 15 per cent H_2SO_4 (by volume). Wash out a Jones reductor[14] with diluted H_2SO_4 (1:19) and water, leaving sufficient water in the reductor to fill to the upper level of the zinc. (These washings should require not more than one or two drops of 0.1 *N* $KMnO_4$ solution to obtain a pink color.) Empty the receiver, and put in it 25 ml. (measured in a graduate) of ferric sulfate solution (Section 29). Reduce the prepared titanium solution as follows:[15]

(*1*) Run 50 ml. of the diluted H_2SO_4 (1:19) through the reductor at such a

[14] Directions for preparing a Jones reductor may be found in Blair, "The Chemical Analysis of Iron," Eighth Edition, pp. 88-89, or Treadwell-Hall, "Analytical Chemistry," Vol. 2, Fifth Edition.

[15] Lundell and Knowles, "The Determination of Titanium by Reduction with Zinc and Titration with Permanganate," *Journal*, Am. Chemical Soc., Vol. 45, p. 2620 (1923).

uniform rate as to require 5 to 10 min. for passage.

(*2*) Follow this with the titanium solution at such a uniform rate as to require 10 min. to pass through the reductor.

(*3*) Wash out with 100 ml. of diluted H_2SO_4 (1:19).

(*4*) Finally run through about 100 ml. of water. Care should be taken that the reductor is always filled with solution or water to the upper level of the zinc. Gradually release the suction, wash thoroughly the glass tube that was immersed in the ferric sulfate solution, remove the receiver, and titrate immediately with 0.1 *N* $KMnO_4$ solution. One milliliter of 0.1 *N* $KMnO_4$ is equivalent to 0.0048 g. of Ti or 0.008 g. of TiO_2. Run a blank determination, using the same reagents, washing the reductor as in the above determination. Subtract this permanganate reading from the original reading and calculate the final reading to titanium dioxide (TiO_2). This will include iron, chromium, arsenic, and any other substance which is reduced by zinc and acid. See Section 38 for reporting TiO_2.

Barium Sulfate (In the case of Titanium-Barium Pigments)

35. Ignite, cool, and weigh the precipitate of $BaSO_4$ obtained in separating the titanium in Section 34.

NOTE.—If sample is impure it may be necessary to purify this precipitate, using appropriate methods.

Calcium Sulfate (In the case of Titanium-Calcium Pigments)

36. Transfer 0.5 g. of the sample to a 250-ml. beaker, add 25 ml. of HCl (1: 1), cover with a watch glass, and boil for several minutes. Dilute to 150 ml. with water, heat to boiling, wash off the cover glass, filter through a 9-cm. filter paper, and wash the paper and residue with hot diluted HCl (1:99) until the washings show no calcium when tested with NH_4OH and $(NH_4)_2C_2O_4$. To the filtrate, add two drops of HNO_3 (sp. gr. 1.42) and boil to insure oxidation of the iron. Add NH_4OH until slightly alkaline, and heat to boiling. Then without filtering, add an excess (30 to 35 ml.) of a hot saturated solution of $(NH_4)_2C_2O_4$, stir, and continue the boiling until the precipitate becomes granular. Let stand on a steam bath for 1 hr., and filter through a 9-cm. filter paper. Wash the beaker and filter with boiling-hot water until 10 ml. of the washings are not decolorized in 2 to 3 min. after adding 0.5 ml. of H_2SO_4, heating to about 70 C., and adding two drops of 0.1 *N* $KMnO_4$ (Section 29). Place the beaker in which precipitation was made under the funnel, pierce the apex of the filter paper with a stirring rod, and wash the precipitate into the beaker with a jet of hot water. Pour 30 ml. of warm diluted H_2SO_4 (1:3) through the filter so that the acid comes in contact with all of the paper, and wash the funnel and filter thoroughly with hot water. Dilute to about 250 ml. with water, heat to about 90 C., and titrate at once with 0.1 *N* $KMnO_4$ solution to a faint pink color that does not disappear in 2 to 3 min. (the temperature of the solution should not be below 60 C. when the end point is reached). Calculate the total calcium to percentage of $CaSO_4$ (1 ml. of 0.1 *N* $KMnO_4$ is equivalent to 0.0068 g. of $CaSO_4$ or 0.0028 g. of CaO. The Fe value of $KMnO_4$ multiplied by 0.502 is equivalent to the CaO value).

Iron Oxide

37. Prepare a standard ferric solution containing 0.00001 g. of Fe per milliliter (Section 29). Weigh a 1-g. portion of the sample and treat as in Section 34. Transfer without filtering to a graduated 200-ml. flask, cool, fill to the mark with

water, mix, let settle, and determine iron colorimetrically as follows: Filter through a dry filter paper, discarding the first 20 ml. Transfer 50 ml. of the clear filtrate to a clean 100-ml. Nessler tube or other comparator. Add a drop or two of 0.1 *N* $KMnO_4$ solution to oxidize any ferrous iron. The faint pink color should persist for at least 5 min. Add 10 ml. of KCNS or NH_4CNS solution (Section 29), dilute with water to 100 ml., and mix thoroughly. Compare the color immediately with a series of reference standards, prepared side by side with the sample, in similar tubes. Prepare the reference standards from the standard ferric solution so as to have a range of from 0.000005 g. Fe to 0.00004 g. Fe (0.5 to 4.0 ml. of the standard solution). Transfer the desired volumes of the standard ferric solution to 100-ml. Nessler tubes containing 50 ml. each of an acid solution (made up by dissolving 8 g. of $(NH_4)_2SO_4$ in water, adding 20 ml. of H_2SO_4 (sp. gr. 1.84), cooling, diluting with water to 200 ml., and mixing), add a drop of 0.1 *N* $KMnO_4$ solution (or sufficient to yield a pink color that will persist for 5 min.), and then 10 ml. of the thiocyanate solution. Finally dilute all standards with water to 100 ml. and mix each thoroughly.

NOTE.—For a single sample it is more convenient to run the standard Fe solution from a burette into a Nessler tube containing 50 ml. of acid solution (made by dissolving 8 g. of $(NH_4)_2SO_4$ in water, adding 20 ml. of concentrated H_2SO_4 (sp. gr. 1.84), cooling, and diluting with water to 200 ml., and mixing), a drop of 0.1 *N* $KMnO_4$, 10 ml. of the thiocyanate solution, and then dilute with distilled water until the depth of the color produced after diluting 100 ml. and mixing, exactly matches that of the sample. From the burette reading calculate the amount of Fe. When using standards, the color comparisons must be made immediately.

Calculations and Report

38. Calculate the total iron found to Fe_2O_3 and report as such. Calculate the TiO_2 equivalent by multiplying the Fe_2O_3 result by the factor 1.003 and subtract this figure from the total TiO_2 as determined in Section 34; report the remainder as TiO_2. Report all results on the dry or moisture-free basis.

CALCIUM PIGMENTS

Whiting, Paris White, Spanish White, Chalk

Qualitative Analysis

39. By the usual methods of qualitative analysis, test the sample for small amounts of insoluble matter (siliceous), iron, aluminum, sulfur, water, or magnesium.

NOTE.—The sample should be approximately 95 per cent $CaCO_3$

Total Soluble Lime[16]

40. Weigh out 0.75 g. of the sample into a small crucible, ignite cautiously to dull redness to destroy organic matter, cool. Transfer to a 400-ml. beaker, add 20 ml. of water, cover, then add 15 ml. of HCl (sp.gr. 1.19) and 3 or 4 drops of HNO_3 (sp.gr. 1.42), and boil until all the soluble matter is dissolved and all the CO_2 expelled. Wash off and remove the cover, dilute the solution to about 150 ml. with freshly boiled water, heat to boiling, and add diluted NH_4OH (sp. gr. 0.96) carefully until a *slight* permanent precipitate forms. Heat to boiling and add 10 ml. of a solution of oxalic acid (10 per cent); stir until the oxides of iron and aluminum are entirely dissolved and only a slight precipitate of calcium oxalate remains. Add 200 ml. of boiling water and sufficient saturated solution of ammonium oxalate (20 to 25 ml.) to precipitate the lime. Boil and stir for a few moments, remove from the heat, let settle, and filter on an 11-cm. filter. Wash ten times with 10 to 15-ml. portions of hot water. Place beaker in which precipi-

[16] Meade, "Portland Cement," 3rd Edition, p. 440 Chemical Publishing Co. (1926).

tation was made under the funnel, pierce apex of filter with stirring rod, and wash precipitate into beaker with hot water. Pour warm diluted H_2SO_4 (1:4) through paper and wash a few times. Add about 30 ml. of the diluted H_2SO_4 (1:4), dilute to about 250 ml., heat to 90 C. and titrate at once with standard $KMnO_4$ solution (solution should not be below 60 C. when end point is reached). The $KMnO_4$ is best standardized against the National Bureau of Standards' standard sample No. 40 of sodium oxalate.[17] Calculate to CaO and $CaCO_3$.

Mixed Calcium and Magnesium Carbonates[18]

41. (*a*) Weigh 1 g. of the finely powdered sample into a small porcelain dish, add 25 ml. of 1 *N* HCl, cover with a watch glass, and when effervescence has ceased, heat to boiling. Cool and titrate with 1 *N* NaOH solution, using methyl orange as indicator.

(*b*) The calculation is as follows:

One gram of CaO = 35.7 ml. of (1 *N*) acid

$$CaCO_3 = CaO \times 1.784$$

Subtract the number of milliliters of NaOH required from 25; the result represents the number of milliliters of (1 *N*) acid corresponding to the $CaCO_3 + MgCO_3$. Multiply the weight of CaO in 1 g. of sample (as found in preceding section on total soluble lime) by 35.7; the product represents the number of milliliters of 1 *N* acid corresponding to the CaO present. Subtract from the total number of milliliters of acid required by $CaCO_3 + MgCO_3$ and multiply the result by 0.042, obtaining weight of $MgCO_3$ in 1 g. of sample. The $MgCO_3$ determined by this process should not differ more than 0.25 per cent from that obtained by more elaborate methods. It is to be noted that this method is a measure of the total alkalinity, and if Ca or Mg are present in other forms than carbonate, a complete analysis would be necessary to give percentages of $CaCO_3$ and $MgCO_3$.

(*c*) Instead of the procedure described in Paragraphs (*a*) and (*b*), Newberry's[19] method for the simultaneous determination of calcium and magnesium carbonates may be used.

Gypsum, Terra Alba, Plaster of Paris

Combined Water and Moisture

42. (*a*) Heat 1 g. of the sample in a covered porcelain crucible on an asbestos plate for 15 min., then heat bottom of the crucible a *dull red* for 10 min. over a bunsen burner, remove cover, and heat for 30 to 40 min. at a slightly lower temperature. Cool and weigh rapidly. Repeat to constant weight.

(*b*) Combined water and moisture may also be determined by heating to constant weight in an air bath at 200 C.

Insoluble Matter

43. Boil 2 to 3 g. of the sample with 20 ml. of HCl (sp. gr. 1.19), a few drops of HNO_3, and about 50 ml. of water. Evaporate to dryness, and boil residue repeatedly with HCl (1:9), decanting through a filter. Finally transfer to the filter, wash with hot water, ignite, and weigh insoluble matter. Test for $BaSO_4$. Make a qualitative examination of the filtrate from the insoluble matter to determine if Al, Fe, or Mg are present in abnormal amounts (should be mainly Ca and SO_3). Test a separate portion of the sample for CO_2; if desired determine as described under Basic Carbonate of Lead in Section 7.

[17] Supplement to National Bureau of Standards *Circular No. 398* (1932).

[18] J. W. Mellor, "A Treatise on Quantitative Inorganic Analysis," p. 522, J. B. Lippincott Co., Philadelphia, Pa. (1913).

[19] Meade "Portland Cement," 3d Edition, p. 440, Chemical Publishing Co. (1926); Mellor, "A Treatise on Quantitative Inorganic Analysis," p. 522, J. B. Lippincott Co., Philadelphia, Pa. (1913); *Cement Engineering News*, Vol. 15, p. 35 (1903).

BARIUM PIGMENTS

Barytes or Barite, "Blanc Fixe"

Microscopic Examination

44. Examine the sample microscopically to determine uniformity of grinding, size, and angularity of particles, crystalline or amorphous. The sample should be at least 95 per cent $BaSO_4$.

Loss on Ignition

45. Ignite 1 g. of the sample for 30 min. Loss may be due to organic matter, moisture, combined water, and CO_2 (and SO_3 from $CaSO_4$ if present). If loss is appreciable, test for CO_2.

Soluble and Insoluble Matter

46. Boil 1 g. of the sample with HCl (1:3), filter, wash with hot water, ignite, and weigh insoluble matter. This may be treated with H_2SO_4 and HF for SiO_2. In the absence of Al_2O_3 (from silicates) residue is considered as $BaSO_4$. Make qualitative tests of filtrate from the insoluble matter for Al, Fe, Ca, Mg, SO_3. Test a separate portion of sample qualitatively for CO_2.

SILICA PIGMENTS

Silica or Silex

47. Silica or silex should be practically pure SiO_2. A qualitative examination will suffice in most cases with a determination of the loss on treating 1 g. of the sample with H_2SO_4 and HF.

China Clay

48. A qualitative analysis of China clay to determine whether the clay is essentially hydrous aluminum silicate is generally all of the chemical tests necessary. Color, fineness, etc., are more important than analysis.

Asbestine

49. Asbestine is tested qualitatively to determine whether material is as represented. Color, fineness, etc., are important. Asbestine should be examined under the microscope to note whether it is fibrous or talcose and whether silica or China clay are admixed. Samples of known origin should be used here as a guide for comparison.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: K58-1941

Standard Methods of

CHEMICAL ANALYSIS OF YELLOW AND ORANGE PIGMENTS CONTAINING CHROMIUM COMPOUNDS, BLUE PIGMENTS, AND CHROME GREEN[1]

A.S.T.M. Designation: D 126 – 36

ADOPTED, 1923; REVISED, 1927, 1935, 1936.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 126; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods cover procedures for the chemical analysis of yellow and orange pigments containing chromium compounds, blue pigments, and chrome green.

Preparation of Sample

2. Dry pigments, if lumpy or not finely ground, shall be ground, to a fine powder for analysis. Large samples may be thoroughly mixed and a representative portion taken and powdered if lumpy or not finely ground. The sample in all cases shall be thoroughly mixed before taking portions for analysis. Extracted pigments shall be ground to a fine powder, passed through a No. 80 (177-micron) sieve[3] to remove any skins, and thoroughly mixed. The weighed portions of extracted pigments should be moistened with a little alcohol before adding reagents for analysis. All samples shall be preserved in stoppered bottles or containers.

YELLOW AND ORANGE PIGMENTS

(Chrome Yellows, American Vermillion, Basic Lead Chromate)

NOTE.—A pure chrome yellow should contain only lead chromate and other insoluble lead compounds.

Added Coloring Matter

3. Boil 2 g. of the sample with 25 ml. of distilled water, let settle, decant the supernatant liquid. Boil the residue with 25 ml. of 95 per cent ethyl alcohol and decant as before. Boil the residue with 25 ml. of chloroform and again decant. If any one of the above solutions is colored, organic coloring matter is indicated. If the solutions should remain colorless, organic colors are probably absent.

NOTE.—If it is desired to test for organic colors resistant to the above reagents, the test

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these methods were published as tentative from 1922 to 1923, being revised in 1923. Editorially revised and rearranged in 1939.

[3] Detailed requirements for this sieve are given in the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

procedures described in the following books may be used, taking into account the nature of the pigment involved: Zerr and Mayer, "Tests for Coal Tar Colors in Aniline Lakes"; Schultz and Julius, "A Systematic Survey of the Organic Coloring Matters"; Mulliken, "Identification of Pure Organic Compounds," *Commercial Dyestuffs*, Vol. III.

Moisture

4. Heat 2 g. of the sample at 105 C. for 2 hr. Report the loss in weight as moisture.

Insoluble Matter

5. Treat 1 g. of the sample with 25 ml. of HCl (sp. gr. 1.19) and boil for from 5 to 10 min. in a covered beaker, adding about six drops of alcohol to the boiling liquid, one at a time. Dilute to 100 ml. with hot water and boil for from 5 to 10 min. (solution should be complete). Filter the hot solution (if insoluble matter is present) and wash with boiling water until washings are free from lead and chlorine. Ignite the insoluble matter, weigh, and examine for SiO_2, $BaSO_4$, and Al_2O_3.

Total Lead

6. Add NH_4OH to the filtrate from the insoluble matter (or the original solution) until a faint precipitate begins to form, then add 5 ml. of HCl (sp. gr. 1.19), dilute to 500 ml., and pass into the clear solution a rapid current of H_2S until all of the lead is precipitated as PbS. Let the precipitate settle, filter, wash with water containing some H_2S. Boil the filter and precipitate with diluted HNO_3 (1:3) until all of the lead has dissolved, filter, and wash thoroughly with hot water. To the filtrate, add 10 ml. of H_2SO_4 (1:1), evaporate until copious fumes of H_2SO_4 are evolved, cool, add about 75 ml. of water and then 75 ml. of 95 per cent ethyl alcohol. Let stand about 1 hr., filter on a Gooch crucible, wash with diluted alcohol, dry, ignite, and weigh as $PbSO_4$. Save the alcoholic filtrate from the $PbSO_4$, evaporate nearly to dryness, and add to the filtrate from the PbS.

Chromium (Iron, Aluminum)

7. (*a*) Heat the filtrate from the PbS (Section 6) to expel H_2S and, if iron is present, add a few drops of HNO_3 and boil about 2 min. Render the solution just alkaline with NH_4OH, boil a few minutes, filter, and wash with hot NH_4Cl solution (2 per cent). (If the sample contains an appreciable amount of zinc, a double precipitation should be made.) In the absence of iron and aluminum this precipitate may be ignited and weighed as Cr_2O_3. If iron and aluminum are present, dissolve the NH_4OH precipitate with hot diluted HCl, washing the paper with hot water. Evaporate to about 100 ml., *cool*, add NH_4OH until alkaline, and then add Na_2O_2 (containing at least 90 per cent of Na_2O_2) in small portions to the cooled solution (10 to 12 C.) until oxidation is complete, keeping the beaker covered. Digest until all of the chromium and aluminum have been dissolved, adding more Na_2O_2 if necessary. Filter off the $Fe(OH)_3$, wash thoroughly with hot water, ignite, and weigh as Fe_2O_3; or, dissolve the precipitate in HCl and determine the Fe content volumetrically. Make up the filtrate from the $Fe(OH)_3$ to 250 ml. in a graduated flask, and mix. Boil an aliquot portion vigorously for 10 to 15 min. to expel H_2O_2, cool, acidify with H_2SO_4, add an excess of standard $(NH_4)_2Fe(SO_4)_2 \cdot 6H_2O$ solution and titrate back with $K_2Cr_2O_7$ solution (0.1 *N*), using $K_3Fe(CN)_6$ as outside indicator. (The CrO_3 may also be determined by acidifying the aliquot portion with acetic acid, precipitating as $PbCrO_4$ or $BaCrO_4$, and finally weighing on a Gooch crucible.) To determine Al_2O_3, make an aliquot portion of the filtrate from the $Fe(OH)_3$ acid with HCl, and then just distinctly alka-

line with NH_4OH, heat to boiling, let settle, filter, wash with hot NH_4Cl solution (2 per cent), ignite, and weigh as Al_2O_3. If iron and aluminum are not to be determined or are present in negligible amounts, the first NH_4OH precipitate may be dissolved in diluted HCl, oxidized with Na_2O_2, boiled vigorously for 10 to 15 min. to expel H_2O_2, cooled, acidified with H_2SO_4, and CrO_3 determined volumetrically.

(*b*) The CrO_3 in the absence of other oxidizing substances, may be determined on 1 g. of the pigment by Schwartz' method[4] or by the persulfate method,[5] as follows: For c. p. chrome yellows use a 0.25-g. sample, for c. p. chrome greens use a 0.5-g. sample, and for commercial chrome greens use a 1-g. sample. Weigh the sample, transfer to a 600-ml. resistant glass beaker,[6] add 25 ml. of H_2SO_4 (sp. gr. 1.84), and fume lightly on a hot plate for 3 or 4 min. Excessive fuming or cooking over a hot flame is neither necessary nor desirable, as it may produce the difficultly soluble anhydrous chromium sulfate (Note 1). The Prussian blue color is destroyed in a few minutes. Cool, cautiously dilute with water to 300 ml. (Note 2), stir, and heat to boiling. To the boiling solution add a small amount of permanganate (0.5 to 1 ml. of $KMnO_4$ (0.1 *N*)) to insure the presence of some manganese. Then add to the boiling solution 10 ml. of $AgNO_3$ solution (2.5 g. in enough water to make 1 liter) and 20 ml. (adding this slowly) of ammonium persulfate solution (4 g. of ammonium persulfate of full strength dissolved in 20 ml. of water). The hot solution should show the usual permanganate color. If this color does not develop, or if it disappears, add more persulfate. When the permanganate color is permanent, continue the boiling (best done on a hot plate) for 10 to 15 min. to destroy excess persulfate. Then add 5 ml. of diluted HCl (1:3) and boil for 5 to 8 min. to reduce the oxidized manganese. The color now should be the usual chromate yellow. Cool to about 20 C. and titrate either electrometrically with $FeSO_4$,[7] or add a measured excess of $FeSO_4$ solution (beyond the deep grass-green color) and titrate back with $KMnO_4$ (0.1 *N*). The first faint permanent darkening of the green color is taken as the end point. Correction for a blank (about 0.2 ml. of $KMnO_4$) due to color, etc., should be made.

NOTE 1.—The important precautions are to avoid overheating with H_2SO_4, and to make sure that the persulfate (solid material) has not deteriorated. It should contain over 90 per cent of the reagent.

NOTE 2.—In commercial chrome greens containing silicates and barium sulfate, filtration to remove the "acid insoluble matter" after fuming the sample with acid and diluting is advisable though not necessary. The end point of the subsequent titration of the clear solution is easier to see, and there is less chance for "bumping" during boiling if these are removed. In the case of commercial greens and yellows, the solution after fuming with H_2SO_4 and diluting could be filtered and the precipitated lead sulfate weighed directly for total lead. This would likewise give a clear filtrate for the chromium titration.

Zinc, Calcium, and Magnesium

8. Precipitate with H_2S any zinc in the filtrate from the first NH_4OH precipitate, filter, wash with diluted $(NH_4)_2S$, dissolve the zinc sulfide in diluted HCl, and determine the Zn content volumetrically by the $K_4Fe(CN)_6$ method. In the filtrate from the zinc sulfide, determine calcium by the oxalate method and magnesium as $Mg_2P_2O_7$.

[4] Fresenius, "Quantitative Chemical Analysis," 6th Edition, Vol. 1, p. 424.

[5] E. F. Hickson, *Circular No. 294*, Scientific Section, Am. Paint and Varnish Manufacturers Assn., November, 1926.

[6] Pyrex glass is very satisfactory for this purpose.

[7] Kelly, *Journal of Industrial and Engineering Chemistry*, Vol. 13, p. 1053 (1921).

Sulfuric Anhydride

9. Heat 1 g. of the sample with 10 ml. of HCl (sp. gr. 1.19) until free chlorine is expelled, add about 300 ml. of water, and boil. Filter off any insoluble matter and wash thoroughly with hot water, heat to boiling, and precipitate with $BaCl_2$ solution in the usual manner. Keep the solution hot while filtering off the $BaSO_4$, and wash with hot water until the washings show no lead or chlorine.

Carbon Dioxide

10. Determine carbon dioxide by the evolution method, using diluted HNO_3 free from oxides of nitrogen.

Water-Soluble Matter

11. Transfer 2.5 g. of the sample to a graduated 250-ml. flask, add 100 ml. of water and boil for 5 min. Cool to room temperature, dilute to the mark with water, mix, and allow to settle. Filter the supernatant liquid through a dry filter paper and discard the first 25 ml. of the filtrate. Transfer 100 ml. of the clear filtrate to a weighed dish, evaporate to dryness on a steam bath, dry for 1 hr. in an oven at 105 to 110 C., cool, and weigh. Calculate the percentage of water-soluble matter.

Calculations

12. Calculate CrO_3 to $PbCrO_4$ and SO_3 to $PbSO_4$ if calcium is absent. If CO_2 is present and calcium and magnesium are absent, calculate to $(PbCO_3)_2 \cdot Pb(OH)_2$. Report any residual Pb as PbO. If calcium is present, calculate to $CaCO_3$ if CO_2 is also present. If calcium and SO_3 are present and CO_2 is absent, calculate to $CaSO_4$. If calcium, CO_2, and SO_3 are present, calculate to $CaCO_3$; any residual calcium is then calculated to $CaSO_4$. Report zinc as ZnO.

BLUE PIGMENTS

(Iron Cyanide Blues (Prussian Blue, Chinese Blue, Antwerp Blue, Milori Blue, Bronze Blue, Steel Blue))

NOTE.—The analysis of these blues, as is generally the case with pigments, does not necessarily give results which can be used to grade samples, the tinting strength and color tests being most important.

Moisture

13. Heat 2 g. of the sample at 105 C. for 2 hr. Report the loss in weight as moisture.

Insoluble Matter

14. Ignite 1 g. of the sample in a porcelain dish at a low temperature, just high enough to decompose the last trace of blue but not high enough to render the iron difficultly soluble in HCl. Cool, add 15 ml. of HCl (sp. gr. 1.19) and a few drops of bromine, cover with a watch glass, and digest on the steam bath. Wash off cover, evaporate to a sirup, add water, boil, filter, wash with hot water, ignite the residue, and weigh. Examine the insoluble residue for silica, barium sulfate, and alumina.

Iron and Aluminum

15. Determine iron and aluminum in the filtrate from the insoluble matter (Section 14) by precipitation with NH_4OH in the usual manner. A double precipitation is desirable. Ignite and weigh $Fe_2O_3 + Al_2O_3$, deduct Fe_2O_3 (calculated from total Fe), and calculate Al_2O_3 to Al.

Calcium

16. Determine calcium in the filtrate from the $Fe_2O_3 + Al_2O_3$ by precipitation with ammonium oxalate. Titrate with $KMnO_4$, or ignite and weigh as CaO. Acidity the filtrate from the calcium oxalate with HCl, dilute to a definite volume, and mix.

Sulfuric Anhydride

17. Determine sulfuric anhydride in an aliquot of the solution, just prepared in Section 16 from the filtrate from the calcium oxalate, as $BaSO_4$ in the usual manner (see Section 9).

Alkali Metal and Alkaline Salts

18. Evaporate an aliquot of the solution, just prepared in Section 16 from the filtrate from the calcium oxalate, with H_2SO_4, ignite (treating with solid ammonium carbonate), and weigh. Determine whether the alkali metal is sodium or potassium and subtract the alkali metal corresponding to the sulfate (SO_4) found. The remainder is alkali combined with the blue and is reported as Na or K.

Total Iron

19. Decompose and dissolve 1 g. of the sample as described in Section 14, reduce, and determine the total iron with $KMnO_4$ or $K_2Cr_2O_7$. There should be not less than 30 per cent, calculated on the basis of the dry pigment.

Total Nitrogen

20. Determine the total nitrogen on 1 g. of the sample by the Kjeldahl-Gunning Method, digesting for at least 2½ hr. The H_2SO_4 should not blacken, as that would indicate organic adulteration.

Water-Soluble Matter

21. Transfer 2.5 g. of the sample to a graduated 250-ml. flask, fill to the mark with water, and stopper. Shake vigorously and let stand at room temperature for not less than 15 hr., shaking vigorously from time to time. Then let settle, filter through a dry filter paper, and discard the first 25 ml. of the filtrate. Transfer 100 ml. of the clear filtrate to a weighed dish, evaporate to dryness on a steam bath, dry for 1 hr. in an oven at 105 to 110 C., cool, and weigh. Calculate the percentage of water-soluble matter.

Added Coloring Matter

22. Determine added coloring matter in accordance with Section 3, omitting the treatment with hot water. Also, test the pigment with diluted HCl (1:1) for ultramarine blue (evolution of H_2S) and carbonates (evolution of CO_2).

Calculations

23. The percentage of Prussian blue may be obtained with sufficient accuracy for commercial purposes by multiplying the percentage of nitrogen by 3.404 or the percentage of ammonia by 2.8.

NOTE.—Some blues, for example, Chinese blue, may contain tin salts. Others may contain manganese or chromium compounds. The presence of these compounds should be determined by a qualitative examination at least.

ULTRAMARINE BLUE

NOTE.—An analysis is of little value for determining the quality of pure ultramarines, but is useful in the identification of foreign admixtures. Practical tests as to the stability and compatability of the pigment in mixtures with other pigments, coloring power, tint, fineness, fastness to light, etc., are more important than chemical analysis.

Moisture

24. Heat 2 g. of the sample at 105 C. for 2 hr., cool, and weigh. Report the loss in weight as moisture.

Silica

25. Treat 1 g. of the sample in a covered dish or casserole with 30 ml. of HCl (1:1), heat until decomposed. Wash off and remove cover, and evaporate to dryness on the steam bath. Moisten with HCl (sp. gr. 1.19) and again evaporate to dryness, add 1 to 2 ml. of HCl (sp. gr. 1.19), let stand about 5 min., add hot water, filter, and wash

the insoluble matter with hot water. If great accuracy is desired, evaporate the filtrate to dryness, take up with HCl and water, filter on a second paper, wash, and add the residue to the main insoluble matter. Ignite the insoluble matter, cool, and weigh. Determine SiO_2 by volatilization with H_2SO_4 and HF. Make a qualitative examination of any residue that may remain.

Alumina

26. Render the filtrate from the silica faintly alkaline with NH_4OH, boil a few minutes, filter, wash with a hot solution of NH_4Cl (2 per cent), ignite, and weigh as $Al_2O_3(+Fe_2O_3)$. For more accurate work, dissolve the $Al(OH)_3$ precipitate in HCl and reprecipitate as above.

Sodium Oxide

27. Acidify the filtrate from the Al_2O_3 with H_2SO_4, evaporate to dryness, ignite (finally adding solid ammonium carbonate), and weigh as Na_2SO_4. Calculate to Na_2O. If calcium is present it should be precipitated with ammonium oxalate in the filtrate from the Al_2O_3, ignited, and weighed as CaO, and the sodium determined in the filtrate from the calcium oxalate, as described.

Total Sulfur

28. Mix 1 g. of the sample with 4 g. of Na_2CO_3 and 4 g. of Na_2O_2 in a nickel crucible, cover with about 1 g. of Na_2CO_3, and fuse, using an aluminum or asbestos shield to prevent the sulfur being taken up from the gas. Dissolve the fused mass in diluted HCl (1:2), filter and wash if necessary (there should be no insoluble residue), precipitate with $BaCl_2$ and determine total sulfur by weighing as $BaSO_4$. Calculate to S.

Sulfur Present as Sulfate

29. Dissolve 1 g. of the sample in diluted HCl (1:1), boil to expel H_2S, and filter if necessary; make the solution faintly alkaline with NH_4OH and just distinctly acid with HCl, and treat with $BaCl_2$ in the usual manner. Calculate $BaSO_4$ to SO_3 and to S.

Sulfur Present as Sulfide

30. Subtract the sulfur present as sulfate from the total sulfur to obtain the sulfur present as sulfide.

COBALT BLUE[8]

NOTE.—This pigment is essentially a compound of the oxides of aluminum and cobalt. Certain shades of ultramarine blue are often sold under the name "cobalt blue."

Moisture

31. Heat 2 g. of the sample at 105 C. for 2 hr. Report the loss in weight as moisture.

Alumina

32. Fuse 1 g. of the sample with 12 to 15 g. of sodium or potassium pyrosulfate, cool, digest with water and HCl, filter, and wash the residue with hot water. Make the filtrate up to 250 ml. in a graduated flask and mix. Ignite the residue, cool, weigh, and examine for SiO_2 and $BaSO_4$. Dilute an aliquot of the filtrate to 200 ml., add 5 g. of NH_4Cl, heat to boiling, and add diluted NH_4OH (1:5) until just distinctly alkaline (a few drops of 0.2 per cent alcoholic solution of methyl red is recommended as indicator). Boil for 1 or 2 min., filter at once, dissolve the precipitate with HCl (1:1), and reprecipitate as before. Filter, wash thoroughly with hot NH_4Cl (or NH_4NO_3) solution (2 per cent), ignite, and weigh as Al_2O_3.

Calcium and Magnesium

33. Unite the filtrates from the Al_2O_3, saturate with H_2S, filter, and determine

[8] "Analysis of Paint and Varnish Products," C. D. Holley, p. 210 (1912).

calcium and magnesium in the filtrate in the usual manner.

Cobalt Oxides

34. Subtract from 100 the constituents determined in accordance with Sections 31 to 33 and report the difference as cobalt oxides, unless a qualitative examination shows the presence of other substances in significant amounts. Should the pigment contain phosphoric acid (or arsenic acid) in more than negligible amounts, these must be removed before determining aluminum, calcium and magnesium.[9]

SUBLIMED BLUE LEAD[10]

Total Lead

35. Digest 1 g. of the sample with 15 ml. of HNO_3 (sp. gr. 1.42) in a covered beaker. Boil the solution until the brown fumes of the oxides of nitrogen have disappeared. Add 6 ml. of H_2SO_4 (sp. gr. 1.84) and again boil until the heavy fumes of SO_3 are evolved. Allow the solution to cool, add 30 ml. of water, and boil. Remove the beaker from the hot plate and allow the solution to stand for from 3 to 4 hr. Filter the solution, washing the precipitate by decantation three or four times and allowing the bulk of the precipitate to remain in the beaker. Place this beaker containing the residual lead sulfate under the funnel used for the filtration, wash the filter paper with 75 ml. of a mixture consisting of 95 ml. of NH_4OH (sp. gr. 0.90), 125 ml. of 80 per cent acetic acid and 100 ml. of water. Follow this washing with 75 ml. of hot water. Boil until all $PbSO_4$ is dissolved. Dilute to 200 ml. with hot water, boil, and then titrate with standard ammonium molybdate solution, using a freshly prepared solution of one part of tannic acid in 300 parts of water as an outside indicator. Run a blank and correct for the same. The ammonium molybdate solution contains 8.67 g. in 1 liter of water and is standardized against pure lead foil, pure PbO, or pure $PbSO_4$.

Total Sulfur

36. Treat 0.5 g. of the sample in a beaker with 10 ml. of water and a few milliliters of bromine water. Boil gently until all the bromine has passed off. Dilute with water, add another portion of bromine water, boil, and continue the treatment until the sediment has become white in color. Add 8 ml. of HNO_3 (sp. gr. 1.42), evaporate the solution until the brown fumes of NO_2 have disappeared, dilute with water and add an excess of Na_2CO_3. Boil gently (covered) for from 10 to 15 min. and let stand for 4 hr. Dilute with hot water, filter, and wash with hot water. Reject the residue. Acidify the filtrate (about 200 ml.) with HCl and add an excess of about 2 ml. of the acid. Boil and add a slight excess of $BaCl_2$ solution (10 per cent). Let stand on a steam bath about 1 hr., filter, wash with hot water, ignite and weigh as $BaSO_4$. Calculate the $BaSO_4$ to S.

Lead Sulfate

37. On a separate sample determine the sulfate directly with Na_2CO_3 as in Section 36, without any preliminary treatment with bromine water or HNO_3.

Lead Sulfite

38. Boil 1.5 g. of the sample with 3 g. of Na_2CO_3, let stand, filter, and wash thoroughly. To the filtrate add 3 ml. of bromine water, heat gently to oxidize the sulfite to sulfate, acidity with HCl, and precipitate with $BaCl_2$ solution. Filter, wash, ignite, and weigh as $BaSO_4$. Deduct the amount present as sulfate

[9] See "Technical Methods of Chemical Analysis," Lunge-Keane, Vol. III, Part II, p. 978, (1914).
[10] "The Chemical Analysis of Lead and Its Compounds," Schaeffer and White pp. 22 to 24.

and calculate the remainder to lead sulfite ($PbSO_3$).

Lead Sulfide

39. Deduct the sulfur present as sulfate and sulfite from the total sulfur and report the difference as lead sulfide (PbS).

Lead Carbonate

40. Determine any CO_2 present by the evolution method, removing any H_2S or SO_2 formed by means of $KMnO_4$ or CrO_3 solution. Calculate to $PbCO_3$.

Lead Oxide

41. Deduct the lead present as sulfate, sulfite, sulfide, and carbonate from the total lead and report the difference as lead oxide (PbO).

Zinc Oxide

42. Boil 1 g. of the sample with a solution of 4 g. of NH_4Cl in 30 ml. of water plus 6 ml. of HCl (sp. gr. 1.19). Dilute to 200 ml. with hot water, add 2 ml. of a saturated sodium thiosulfate solution, and titrate with a standard solution of potassium ferrocyanide, using a 5 per cent solution of a uranium nitrate as an outside indicator. Report as ZnO.

Carbon and Volatile Matter

43. Ignite a weighed portion of the sample in a partially covered crucible at a low red heat for 2 hr., cool, and weigh. Report the loss in weight as carbon and volatile matter.

CHROME GREEN

NOTE.—A pure chrome green should contain only Prussian blue and pure chrome yellow. A microscopic examination should be made to determine whether the green is a combined precipitation product, which is of the greater value, or one mixed after separate precipitation. A good green will show the presence of green and blue particles, while a poor green will show yellow and blue particles mixed with green.

Moisture

44. Heat 2 g. of the sample at 105 C. for 2 hr. Report the loss in weight as moisture.

Insoluble Matter

45. Heat gently 1 g. of the sample in a small porcelain dish until the blue color has been decomposed. The heating should be carried out very carefully so as not to render the iron difficulty soluble. (With some very pure chrome greens it may be advantageous to mix the sample with two to five times its weight of pure $BaSO_4$ before igniting.) Let cool, transfer to a beaker, and determine insoluble matter as outlined in Section 5 for Yellow Pigments.

Lead

46. Determine lead in the filtrate from the insoluble matter determination (Section 45) in accordance with Section 6 for Yellow Pigments.

Iron, Aluminum, and Chromium

47. Determine iron, aluminum, and chromium in the filtrate from the lead determination (Section 46) in accordance with Section 7 for Yellow Pigments, making a double precipitation.

Zinc, Calcium, and Magnesium

48. Determine zinc, calcium, and magnesium in the filtrate from the iron, aluminum, and chromium determination (Section 47) in accordance with Section 8 for Yellow Pigments.

Carbon Dioxide

49. Determine CO_2 by the evolution method, using diluted HNO_3 (1:5).

Sulfuric Anhydride

50. Heat gently 1 g. of the sample as in Section 45, cool, transfer to a beaker, add 30 ml. of HCl (sp. gr. 1.19), cover, and heat on a steam bath for about 30

min. (in some cases, the iron compounds will go into solution more readily by letting the solution stand for some time at room temperature and then heating). Wash the cover, add 50 ml. of boiling water, boil for 5 min., filter, render the filtrate faintly alkaline with NH_4OH, then slightly acid with HCl, heat to boiling, and precipitate with $BaCl_2$ (15 ml. of 10 per cent solution) in the usual manner, boiling about 10 min. Filter, wash with hot water, ignite, and weigh the $BaSO_4$.

Nitrogen

51. Determine nitrogen on 1 g. of the sample by the Kjeldahl-Gunning method, digesting for at least 2½ hr.

Water-Soluble Matter

52. Make the test for water-soluble matter in accordance with Section 21.

Added Coloring Matter

53. Determine added coloring matter in accordance with Section 3. Also, test the pigment with diluted HCl (1:1) for the presence of ultramarine blue (evolution of H_2S).

CHROME OXIDE GREEN

NOTE.—Chrome oxide green is practically pure chromic oxide (Cr_2O_3) without any admixture.

Moisture

54. Determine moisture in accordance with Section 44.

Water-Soluble Matter

55. Determine water-soluble matter in accordance with Section 11.

Total Chromium

56. *Special Solutions Required:* (*a*) *Standard Ferrous Solution.*—Dissolve 39.25 g. of ferrous ammonium sulfate crystals in 950 ml. of distilled water and add 50 ml. of H_2SO_4 (sp. gr. 1.84).

(*b*) *Decinormal Potassium Permanganate Solution.*—Dissolve 3.2 g. of pure $KMnO_4$ in 1 liter of distilled water, let stand 8 to 14 days, siphon off the clear solution (or filter through asbestos), and standardize as follows: In a 400-ml. beaker dissolve 0.20 to 0.25 g. (accurately weighed) of pure $K_2Cr_2O_7$ crystals in about 300 ml. of water and add 20 ml. of H_2SO_4 (1:1). Add a measured excess of $FeSO_4$ solution (0.1 *N*) and titrate back with the $KMnO_4$ solution which is being standardized. Now take the same volume of $FeSO_4$ solution and dilute to 300 ml. and titrate with the $KMnO_4$ solution. The difference between the two titrations represents the ferrous sulfate that was oxidized by the $K_2Cr_2O_7$ in terms of milliliter readings of $KMnO_4$. The weight of pure $K_2Cr_2O_7$ divided by the milliliter readings of $KMnO_4$ (by difference) gives the grams of $K_2Cr_2O_7$ equivalent to 1 ml. of $KMnO_4$. One milliliter of the $KMnO_4$ solution should be equal to about 0.00173 g. of chromium.

57. *Procedure.*— Mix thoroughly about 0.25 g. (accurately weighed) of the sample with approximately 5 g. of sodium peroxide (containing at least 90 per cent Na_2O_2) in a 30-ml. pure iron crucible (ordinary iron crucibles may contain some chromium and manganese) and fuse carefully over a gas flame. Fusion at a low red heat for 5 min., after the mass has melted, will insure complete decomposition. Allow the crucible to partly cool, and while tightly covered, tap on an iron plate to loosen the fusion in a solid cake. Place the cake in a covered beaker and dissolve with 200 ml. of warm water. Rinse the crucible and cover with warm water, adding to the main portion. Add approximately 1 g. of Na_2O_2 and boil the solution vigorously for 10 min. to destroy excess peroxide. If excess peroxide is

not destroyed, some of the chromium will be reduced when the solution is acidified. Cool, and acidify with H_2SO_4 (1:1) (about 50 ml.). A clear solution with a few scales of magnetic oxide should result. Immediately cool again, add 5 ml. of H_3PO_4 (sirup, sp. gr. 1.7), transfer to an 800-ml. beaker, and dilute with cold water to 500 ml. Add an accurately measured excess (about 100 ml.) of approximately 0.1 *N* ferrous ammonium sulfate to the solution while stirring. When reduction is complete a deep pure green color should develop, and a drop of the liquid when tested with a drop of diluted potassium ferricyanide solution should give a blue color. Set the beaker about 0.5 in. above a white tile to secure good light. Determine the excess of ferrous solution by titration with a standard solution of $KMnO_4$ (approximately 0.1 *N*). Take the first faint permanent darkening of the clear green color as the end point. Addition of more $KMnO_4$ increases the depth of the color which finally becomes purple. The end point is quite sharp and well defined, but requires some practice. In exactly the same manner carry through a blank determination, starting with a peroxide fusion. Finally, add the exact volume of ferrous solution as was used in the determination and titrate back with the $KMnO_4$ solution. The difference between the volumes required in the two titrations represents the chromium in the sample, and the percentage is found by multiplying this value by 400 times the chromium titer of the $KMnO_4$ solution.

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Standard Methods of

CHEMICAL ANALYSIS OF YELLOW, ORANGE, RED, AND BROWN PIGMENTS CONTAINING IRON AND MANGANESE[1]

A.S.T.M. Designation: D 50 – 36

ADOPTED, 1918; REVISED, 1927, 1933, 1936.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 50; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods cover procedures for the chemical analysis of yellow, orange, red, and brown pigments containing iron and manganese.

Preparation of Sample

2. Dry pigments, if lumpy or not finely ground, shall be ground to a fine powder for analysis. Large samples may be thoroughly mixed and a representative portion taken and powdered if lumpy or not finely ground. The sample in all cases shall be thoroughly mixed before taking portions for analysis. Extracted pigments shall be ground to a fine powder, passed through a No. 80 (177-micron) sieve[3] to remove any skins, and thoroughly mixed. The weighed portions of extracted pigments should be moistened with a little alcohol before adding reagents for analysis. All samples shall be preserved in stoppered bottles or containers.

INDIAN REDS, RED OXIDES (PRINCE'S METALLIC, TUSCAN RED)

Loss at 100 C.

3. Heat 2 g. at 100 C. in a steam-jacketed oven at atmospheric pressure for 3 hr., or to constant weight.

Loss on Ignition

4. Ignite a portion in a covered porcelain crucible to constant weight. This may include combined water, CO_2, organic matter, and some SO_3 if much $CaSO_4$ is present. CO_2 may be determined on a separate portion of the sample if desired.[4]

Free Acid or Alkali

5. Boil 10 g. of the sample with 100 ml. of water; filter, and wash. Test

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these methods were published as tentative from 1917 to 1918. Editorially revised and rearranged in 1939.

[3] Detailed requirements for this sieve are given in the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

[4] It is inadvisable to use platinum unless it is known that attacking substances are absent.

filtrate with litmus paper; if acid, titrate with standard alkali and methyl orange and calculate to the equivalent of H_2SO_4. If alkaline, titrate with acid and calculate to the equivalent of Na_2O. Test filtrate for alkali salts and alkaline earths.

Added Coloring Matter

6. Boil 2 g. of the sample with 25 ml. of distilled water, let settle, decant the supernatant liquid. Boil the residue with 25 ml. of 95 per cent ethyl alcohol and decant as before. Boil the residue with 25 ml. of 1 *N* alcoholic NaOH and again decant. Boil another 2 g. portion of the sample with 25 ml. of chloroform, let settle, and decant the supernatant liquid. If any one of the above solutions is colored, organic coloring matter is indicated (Note 1). If the solutions should remain colorless, organic colors are probably absent.

NOTE 1.—With this class of pigments the presence of an organic color may often be noted by the characteristic odor given off on ignition.

NOTE 2.—If it is desired to test for organic colors resistant to the above reagents, the test procedures described in the following books may be used, taking into account the nature of the pigment involved: Zerr and Mayer, "Tests for Coal Tar Colors in Aniline Lakes"; Schultz and Julius, "A Systematic Survey of the Organic Coloring Matters"; Mulliken, "Identification of Pure Organic Compounds," *Commercial Dyestuffs*, Vol. III.

Insoluble Matter, Iron Oxide, etc.

7. (*a*) Digest 2.5 g. of the sample (previously roasted at a low temperature if much organic matter is present; if very low in carbonaceous matter a little $KClO_3$ or $NaClO_3$ may be used in effecting solution) with 25 ml. of HCl (adding a little HNO_3 or chlorate, if not already added), wash off cover, and evaporate to dryness. Take up with HCl and water, filter, wash with diluted HCl (1:99) and cold water. Make the filtrate up to 500 ml., mix, and examine as below.[5] Ignite the residue and weigh as "insoluble matter";[6] if this contains $BaSO_4$ it may be determined by fusing with six times its weight of Na_2CO_3, cooling, digesting with hot water, filtering, and washing the residue with hot water until free of sulfate.

(*b*) Remove the filtrate and place the beaker used for the digestion underneath the funnel, pierce the filter with the glass rod, and wash the residue with a little water into the beaker; then pour hot diluted HCl (1:1) over paper, and finally wash with hot water. If necessary add more HCl to the beaker to dissolve the $BaCO_3$. Heat to boiling, add diluted H_2SO_4 in slight excess, and let stand about 1 hr. on a steam bath. Filter, wash, dry, ignite, and weigh as $BaSO_4$. (This subtracted from total insoluble matter will give "insoluble siliceous matter," if it is desired to so report.) If it is desirable to analyze the insoluble siliceous matter, this may be done by the usual methods for silicate analysis, but the results should be reported as a separate analysis.

(*c*) For the determination of iron, place 100 ml. of the first filtrate in a flask, add about 3 g. of granulated zinc, put a funnel into the neck of the flask, heat when the action slackens; if basic salts separate out add a few drops of HCl. When the reduction is complete, add 30 ml. of H_2SO_4 (1:2) and, as soon as the residual zinc is dissolved, wash down the funnel inside and out and the neck of the flask with a fine jet of water, filling the flask (1000 ml.) about two-thirds full. Cool in water and add 10 ml. of "titrating solution" (made by dissolving 160 g. of manganese sulfate in water, diluting to 1750 ml., adding 330 ml. of H_3PO_4 (sp. gr. 1.72) and 320 ml.

[5] For more exact work this filtrate should be evaporated to dryness and SiO_2 removed.

[6] If the insoluble contains appreciable amounts of Fe it will be necessary to fuse it with Na_2CO_3 or potassium pyrosulfate ($K_2S_2O_7$) to determine total Fe in samples.

of H_2SO_4 (sp. gr. 1.84)), and titrate with $KMnO_4$ (5.659 g. per liter) that has been standardized against the National Bureau of Standards' standard sample No. 40 of sodium oxalate. Run a blank on the zinc, correct for same, and calculate total iron as Fe_2O_3. Instead of adding the zinc to the solution, the reduction may be effected in a zinc reductor.[7]

(*d*) The Fe_2O_3 may also be determined by the $K_2Cr_2O_7$ method.[8]

Lime

8. (*a*) Dilute an aliquot of 100 ml. of the original solution to about 200 ml., add 10 ml. of HCl (sp. gr. 1.19), make alkaline with NH_4OH, add 2 or 3 ml. of bromine water, and boil until the excess NH_3 is expelled. Let settle, wash by decantation, redissolve in HCl, and reprecipitate with NH_4OH and bromine water. (Precipitate equals $Fe_2O_3 \cdot Al_2O_3 \cdot TiO_2 \cdot P_2O_5 \cdot MnO_2$.) This precipitate may be ignited and weighed if desired.

(*b*) To the combined filtrates add a few drops of NH_4OH, heat to boiling, and add an excess of saturated ammonium-oxalate solution. Continue the boiling until the precipitate becomes granular, let stand about 30 min., filter, and wash with hot water until free of ammonium oxalate.[9] Place the beaker in which precipitation was made under the funnel, pierce the apex of the filter with a stirring rod, and wash precipitate into beaker with hot water. Pour warm diluted H_2SO_4 (1:4) through paper and wash a few times. Add about 30 ml. of H_2SO_4 (1:4), dilute to about 250 ml., heat to 90 C., and titrate at once with standard $KMnO_4$ solution (solution should not be below 60 C. when end point is reached). Calculate to CaO. (The Fe value of $KMnO_4 \times 0.502$ = CaO.) The calcium oxalate precipitate may be ignited to constant weight as CaO. If desired, magnesia may be determined as $Mg_2P_2O_7$ in the usual manner in the filtrate from the calcium oxalate.[10]

Soluble Sulfates

9. Treat 1 g.[11] of the sample (roasted gently if much organic matter is present) with 30 ml. of HCl (sp. gr. 1.19), boil 10 min., add about 50 ml. of water, boil, filter, and wash with hot water. Heat the solution to boiling, add NH_4OH, filter, and wash a few times with hot water. Dissolve precipitate in hot diluted HCl (1:1), reprecipitate with NH_4OH, wash well with hot water. Render united filtrates just distinctly acid with HCl, boil, add drop by drop, while stirring, a $BaCl_2$ solution (10 per cent) in excess, boil about 10 min., filter on a Gooch crucible, wash with hot water, ignite, and weigh as $BaSO_4$. Calculate to SO_3 or $CaSO_4$.

Total Sulfur

10. Treat 5 g. of the sample in a covered porcelain dish with 50 ml. of aqua regia (1 HNO_3:9 HCl) and evaporate to dryness on steam bath. Add 20 ml. of HCl (sp. gr. 1.19) and about 250 ml. of water, make double NH_4OH precipitation. Determine $BaSO_4$ as described in Section 9.

OCHERS

Procedure

11. Using 1 g. of the sample or an aliquot corresponding to this weight,

[7] Lord and Demorest, "Metallurgical Analysis," 1913, pp. 28–29.

[8] *Ibid.*, pp. 21–26.

[9] For more exact work this precipitate should be dissolved in HCl and the calcium oxalate reprecipitated as above.

[10] If desired, a direct determination of Al_2O_3 may be made on an aliquot of the solution or on the HCl solution of the NH_4OH precipitate by Peters' phosphate method (this will include titantic acid) as described by Blair, "The Chemical Analysis of Iron," and Philips, "Methods of Iron Analysis Used in the Pittsburgh District."

[11] If low in soluble sulfates, use a larger portion of the sample.

determine the loss at 100 C., loss on ignition, insoluble matter, total or soluble iron, alumina, lime, and sulfur as described in Sections 3, 4, 7, 8, 9, and 10. Test for solubility in water and reaction to litmus paper.

Lead

12. If present, the lead is removed in the above scheme by nearly neutralizing the filtrate from the insoluble matter with NH_4OH, cooling, and passing in H_2S to precipitate PbS. Filter, wash with water containing H_2S, dissolve PbS in hot diluted HNO_3, add 10 ml. of H_2SO_4 (sp. gr. 1.84), evaporate until SO_3 is evolved, cool, dilute to 200 ml., let stand a few hours, filter on a Gooch crucible, wash with H_2SO_4 (1 per cent), ignite, and weigh as $PbSO_4$. Calculate to PbO or Pb. Heat the filtrate from the PbS to expel H_2S, oxidize with a little HNO_3, and dilute to volume if working on more than 1 g.

Iron

13. The iron is best determined in an aliquot by the $K_2Cr_2O_7$ method. Treat another aliquot with NH_4OH, the precipitate contains $Al_2O_3 \cdot Fe_2O_3 \cdot Cr_2O_3 \cdot P_2O_5 \cdot TiO_2$. Lime and MgO may be determined in the filtrate.

Chromic Oxide

14. (*a*) Dissolve the NH_4OH precipitate in hot diluted HCl, wash paper with hot water, *cool*, oxidize with Na_2O_2, boil vigorously for 10 to 15 min. to expel H_2O_2, and cool. Wash off cover glass, dilute to about 150 ml., and acidify with H_2SO_4. Add a measured excess of ferrous ammonium sulfate solution (12.4 g. of $(NH_4)_2Fe(SO_4)_2 \cdot 6H_2O$ and 50 ml. of H_2SO_4 (sp. gr. 1.84), diluted with water to 1 liter) and titrate back with standard $K_2Cr_2O_7$ solution, using $K_3Fe(CN)_6$ solution as an outside indicator. Titrate the $(NH_4)_2Fe(SO_4)_2 \cdot 6H_2O$ solution with the $K_2Cr_2O_7$ solution to determine its value in terms of the latter. The Fe value of the $K_2Cr_2O_7$ solution $\times$ 0.597 = CrO_3.

(*b*) Or, moisten 1 g. of the sample with water, add 5 ml. of HCl (sp. gr. 1.19), boil a few minutes, *cool*, add Na_2O_2 in excess, boil to expel H_2O_2, cool, wash off the cover glass, dilute, acidify with H_2SO_4, and titrate CrO_3 as described in Paragraph (*a*).

(*c*) *Calculation.*—Calculate the chromic oxide as follows:

$$CrO_3 = A \times 0.597$$

where:

A = the Fe value of the $K_2Cr_2O_7$ solution.

VENETIAN RED

Procedure

15. Analyze the sample as described in Sections 3 to 10. Insoluble matter may be treated with HF and H_2SO_4 to determine SiO_2 by loss, if desired.

SIENNAS AND UMBERS

Procedure

16. After gently roasting the sample to destroy organic matter, analyze it as described in Sections 3 to 10.

Manganese

17. *Special Solutions Required*: (*a*) *Standard $KMnO_4$.*—Dissolve 1 g. of $KMnO_4$ in a liter of water. The Fe value of this solution $\times$ 0.197 = Mn. This solution may be standardized against the National Bureau of Standards' Standard Sample No. 40 of sodium oxalate (using about 0.05 to 0.1 g.).[12] Weight of $Na_2C_2O_4 \times 0.164$ = Mn.

(*b*) *Standard Ferrous Ammonium Sulfate Solution.*—Twelve grams of

[12] W. Blum, "Original Communications," Eighth International Congress of Applied Chemistry, Vol. 1, pp. 61–85.

$Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$, 25 ml. of H_2SO_4 (sp. gr. 1.84), and 25 ml. of H_3PO_4 (sp. gr. about 1.7) are made up to 1 liter with water. The value of this solution should be determined against the $KMnO_4$ each day as follows: Measure into a 200-ml. Erlenmeyer flask 50 ml. of diluted HNO_3 (1:3), cool, add a little bismuthate, dilute with 50 ml. of HNO_3 (3:97), filter by suction through an asbestos felt into a 300-ml. Erlenmeyer flask, and wash with 50 ml. of HNO_3 (3 per cent). Run in 25 ml. of the ferrous solution and titrate with $KMnO_4$ solution. Instead of titrating the permanganic acid formed by the bismuthate with the ferrous solution and then titrating back with $KMnO_4$, a direct titration with standard sodium arsenite solution may be made.[13]

Manganese

18. *Procedure.*—Manganese is determined by the bismuthate method.[14] Ignite gently (to destroy organic matter) 1 g. of the sample in a platinum dish, cool, add 10 ml. of water, 4 ml. of H_2SO_4 (sp. gr. 1.84), and about 20 ml. of HF (if necessary, add a little sulfurous acid). Evaporate until the H_2SO_4 fumes freely, cool, and dissolve in 25 ml. of diluted HNO_3 (1:3). If no appreciable residue remains, transfer to a 100-ml. volumetric flask, using 25 ml. of diluted HNO_3 (1:3) to rinse the dish, dilute to the mark with water, mix thoroughly. If there is an appreciable residue, filter on a small filter, wash with water, ignsit residue in a platinum crucible, and fuee with a little sodium or potassium pyrosulfate. Dissolve in water, with the addition of a little HNO_3, add to the main filtrate, evaporate nearly to dryness, take up in diluted HNO_3 (1:3) and transfer to the flask as before. Pipette an aliquot of 10 ml. into a 200-ml. Erlenmeyer flask, add 30 ml. of water and 10 ml. of HNO_3 (sp. gr. 1.42); add about 0.5 g. of sodium bismuthate, heat for a few minutes, or until the pink color has disappeared with or without the precipitation of MnO_2. Add a few small crystals of $NaNO_2$ or KNO_3 to dissolve the MnO_2 and boil the solution several minutes to expel nitrous fumes (a little Na_2CO_3 will aid this). Add water to bring the volume up to 50 ml. and cool to about 15 C.; add about 0.5 g. of bismuthate and shake the flask well. Add 50 ml. of water containing 30 ml. of HNO_3 (sp. gr. 1.42) to the liter, filter by suction through an asbestos felt into a 300-ml. Erlenmeyer flask, and wash with 50 to 100 ml. of the same acid. Run in a measured volume of standard ferrous ammonium sulfate solution and titrate to a faint pink color with standard $KMnO_4$ solution. The number of milliliters of the $KMnO_4$ solution obtained, subtracted from the number corresponding to the volume of ferrous solution used, will give the volume of $KMnO_4$ equivalent to the manganese in the sample, which, multiplied by the value of the $KMnO_4$ in Mn, gives the weight of manganese in the portion of sample used.

[13] Lord and Demorest, "Metallurgical Analysis," 1913, p. 82.
[14] Blair, "The Chemical Analysis of Iron."

Standard Methods of

CHEMICAL ANALYSIS OF ZINC DUST (METALLIC ZINC POWDER)[1]

A.S.T.M. Designation: D 521 - 40

ADOPTED, 1940.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 521; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods cover procedures for the chemical analysis of metallic zinc powder in the form commercially known as zinc dust for use as a pigment in paints.

Treatment of Sample

2. The sample in all cases shall be thoroughly mixed before taking portions for analysis. All samples shall be preserved in stoppered bottles or containers.

NOTE.—Caution should be observed in protecting the sample from oxidation.

MOISTURE AND OTHER VOLATILE MATTER

Procedure

3. Weigh 2 g. of the sample in a tared moisture dish. Dry for 2 hr. at 105 to 110 C., cool, and weigh. Calculate the percentage of moisture and other volatile matter, as follows:

$$M = \frac{L \times 100}{W}$$

where:

M = percentage of moisture and other volatile matter,
L = loss in weight, and
W = weight of sample.

TOTAL ZINC

Reagent

4. *Potassium Ferrocyanide Solution.*—Dissolve 22 g. of $K_4Fe(CN)_6 \cdot 3H_2O$ in water and dilute to 1000 ml. To standardize, transfer about 0.2 g. (accurately weighed) of pure metallic zinc or freshly ignited pure zinc oxide to a 400-ml. beaker. Dissolve in 10 ml. of HCl (sp. gr. 1.19) and 20 ml. of water. Drop in a small piece of litmus paper, add NH_4OH until slightly alkaline, then add HCl until just acid, and then 3 ml. more of HCl. Dilute to about 250 ml. with hot water, and heat nearly to boiling. Run in the $K_4Fe(CN)_6$ solution slowly from a burette, while stirring constantly, until a drop tested on a white porcelain plate with a drop of the uranyl indicator shows a brown tinge after standing 1 min. Run a blank using the same amounts of reagents and water as in the standardization. Subtract the amount of $K_4Fe(CN)_6$ solution required for the blank from the amounts used in stand-

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these methods were issued as tentative from 1939 to 1940, being revised in 1940.

ardization and in titration of the sample. The standardization must be made under the same conditions of temperature, volume, and acidity as obtain when the sample is titrated. Calculate the strength of the $K_4Fe(CN)_6$ solution in terms of grams of zinc as follows:

$$1 \text{ ml. of } K_4Fe(CN)_6 = \frac{\text{wt. of zinc in grams}}{\text{ml. of } K_4Fe(CN)_6}$$

Procedure

5. Weigh accurately about 0.25 g. of the sample, transfer to a 400-ml. beaker, moisten with alcohol, dissolve in 10 ml. of HCl (sp. gr. 1.19) and 20 ml. of water, and titrate with standard $K_4Fe(CN)_6$ following the procedure used in standardizing that solution as described in Section 4.

Calculation

6. Calculate total zinc as zinc oxide according to the following formulas:

$$\text{Total Zinc, per cent} = \frac{a \times b \times 100}{W}$$

$$\text{ZnO, per cent} = \text{Zn, per cent} \times 1.2447$$

where:

a = milliliters of $K_4Fe(CN)_6$,
b = grams of zinc per milliliter, and
W = weight of sample.

METALLIC ZINC

Reagents

7. (*a*) *Potassium Permanganate Solution (0.1 N).*—Dissolve 3.2 g. of $KMnO_4$ in 1000 ml. of water. Allow to age for at least 10 days, and filter through purified asbestos. Standardize against the National Bureau of Standards' standard sample No. 40 of sodium oxalate as follows: Transfer 0.3 g. of standard sodium oxalate (dried at 105 C.) to a 600-ml. beaker. Add 250 ml. of diluted H_2SO_4 (5:95) which has been previously boiled for 10 to 15 min. and cooled to 27 ± 3 C. Stir until the oxalate has dissolved. Add 39 to 40 ml. of $KMnO_4$ (0.1 *N*) (0.3 g. of sodium oxalate requires 44.78 ml. of 0.1 *N* $KMnO_4$) at a rate of 25 to 35 ml. per min., while stirring slowly. Let stand until the pink color disappears (about 60 sec.). (If the pink color should persist because of the addition of an excess of the permanganate solution, discard and begin again, adding a few milliliters less of the $KMnO_4$ solution.) Heat to 55 to 60 C., and complete the titration by adding permanganate until a faint pink color persists for 30 sec. Add the last 0.5 to 1 ml. drop by drop with particular care to allow each drop to become decolorized before the next is introduced. Determine the excess of permanganate required to impart a pink color to the solution by matching the color in another beaker containing the same amount of specially treated diluted H_2SO_4 (5:95) at 55 to 60 C. This correction usually amounts to 0.03 to 0.05 ml.

(*b*) *Sodium Acetate Solution (20 per cent).*

(*c*) *Ferric Chloride Solution.*—Prepare a solution containing 20 g. of $FeCl_3 \cdot 6H_2O$ and 20 ml. of the $NaC_2H_3O_2$ solution per 100 ml. It is advisable to make up only one day's supply at a time (50 ml. for each determination).

(*d*) *Zimmerman-Reinhardt Solution.*—Prepare a solution containing 67 g. of $MnSO_4 \cdot 4H_2O$, 130 ml. of H_2SO_4 (sp. gr. 1.84), and 138 ml. of H_3PO_4 (sp. gr. 1.7) per liter.

Procedure[3]

8. Weigh accurately approximately 0.2 g. of the sample, transfer immediately to a dry, 600-ml., heavy-wall Erlenmeyer flask, and add 50 ml. of the $FeCl_3$ solution. Tightly stopper the flask, and agitate constantly for approximately 15 min. As soon as the zinc dust is all

[3] This is a rapid method intended for routine analysis. The results are inclined to be somewhat low, and for highest accuracy and particularly for referee tests, the hydrogen evolution method should be used. See *Proceedings*, Am. Soc. Testing Mats., Vol. 18, Part II, p. 220 (1918).

dissolved, add 50 ml. of the Zimmerman-Reinhardt solution and 250 ml. of water, and titrate with the standard $KMnO_4$ solution. Correct for a blank titration (usually 0.1 to 0.2 ml.). One milliliter of 0.1 *N* $KMnO_4$ is equivalent to 0.00327 g. of zinc.

Calculation

9. Calculate metallic zinc according to the following formula:

$$\text{Metallic zinc, per cent} = \frac{\text{ml. of } KMnO_4 \times 0.00327}{\text{wt. of sample}} \times 100$$

NOTE.—If preferred, the reduced iron may be titrated with a 0.1 *N* standard solution of $K_2Cr_2O_7$ using sodium diphenylamine sulfonate in the presence of H_3PO_4 as an internal indicator.[4]

ZINC OXIDE

Calculation

10. Subtract the percentage of metallic zinc (Section 9) calculated to zinc oxide from the percentage of total zinc as zinc oxide (Section 6). The remainder is the percentage of zinc oxide present.

[4] See "Textbook of Quantitative Inorganic Analysis" by Kolthoff and Sandell, 1936, p. 579.

Standard Methods of

CHEMICAL ANALYSIS OF ZINC YELLOW PIGMENT (ZINC CHROMATE YELLOW)[1]

A.S.T.M. Designation: D 444 – 39

ADOPTED, 1939.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 444; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

1. These methods cover procedures for the chemical analysis of the pigment known commercially as "zinc yellow," or "zinc chromate yellow" (Notes 1, 2, and 3).

NOTE 1.—According to the work of Denslow, it appears probable that zinc yellow is a definite salt, the "oxide formula" of which would be $K_2O \cdot 4ZnO \cdot 4CrO_3 \cdot 3H_2O$.

NOTE 2.—For ordinary routine work, the following determinations should be sufficient: combined water, chromium (CrO_3), zinc (ZnO), total alkali metals (K_2O), and sulfates (SO_3). These should give a summation close to 100 per cent.

NOTE 3.—In commercial zinc yellow pigments, using the procedures described in these methods, the percentage of zinc calculated as ZnO generally varies between 37 and 40. The chromium as CrO_3 generally varies between 41 and 43 per cent. The sulfates as SO_3 generally vary between 1.4 and 3.3 per cent. The combined water generally varies between 6 and 7 per cent. The alkali metals expressed as K_2O generally vary between 10 and 12 per cent.

Preparation of Sample

2. The dry pigment, if lumpy or not finely ground, shall be ground to a fine powder for analysis. Large samples may be thoroughly mixed and a representative portion taken and powdered, if lumpy or not finely ground. The sample in all cases shall be thoroughly mixed before taking portions for analysis. Extracted pigments shall be ground to a fine powder, passed through a No. 80 (177-micron) sieve[3] to remove any skins, and thoroughly mixed. The weighed portions of extracted pigments should be moistened with a little alcohol before adding reagents for analysis. All samples shall be preserved in stoppered bottles or containers.

Moisture

3. Weigh 6 g. of the sample in a tared moisture dish. Dry the sample over night at 110 C. Cool, and weigh the

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these methods were published as tentative from 1937 to 1939, being revised in 1938 and 1939.

[3] Detailed requirements for this sieve are given in the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

sample. Calculate the percentage of moisture as follows:

$$\text{Moisture, per cent} = \frac{\text{loss of wt.}}{\text{wt. of sample}} \times 100$$

Combined Water and Moisture

4. Weigh 3 g. of the sample and transfer to a porcelain boat. Introduce the boat with the charge into a silica combustion tube of an electrically-heated combustion furnace (of the type used for the determination of carbon in steel by direct combustion).[4] Place the boat at the center of the combustion tube maintained at a temperature of 1000 C. A current of pure dry air drawn through the tube sweeps the evolved moisture through a train of absorption tubes containing H_2SO_4 or dry $CaCl_2$. The weight increase of the absorption tube represents the "combined water" (including moisture). Report the results as combined water.

NOTE 1.—Loss on ignition of the pigment does not suffice for the determination of combined water in zinc chromates.

NOTE 2.—Traces of oil in the pigment should first be removed with ether.

Solution of Sample for Analysis

5. Dissolve 4 g. of the sample in 50 ml. of cold 6 *N* H_2SO_4 (160 ml. of H_2SO_4 (sp. gr. 1.84) per liter of solution). It should dissolve completely at this stage. Transfer the solution to a 500-ml. calibrated flask and dilute to 500 ml. The solution should be approximately 0.1 *N* in terms of chromic acid.

Chromium

6. (*a*) *Special Solution Required.*—Standard sodium thiosulfate ($Na_2S_2O_3$) solution (0.1 *N*).

(*b*) *Procedure.*—To a 400-ml. beaker containing 200 ml. of water, add 2 g. of KI (solid) and 5 ml. of H_2SO_4 (sp. gr. 1.84). (Final acidity should be approximately 5 ml. of acid per 100 ml. of solution.) Pipette a 25-ml. aliquot from the zinc chromate – sulfuric acid solution as made up in the zinc oxide determination into a 500-ml. glass-stoppered Erlenmeyer iodimetric flask or other suitable glass-stoppered bottle, containing 200 ml. of diluted sulfuric acid (25 ml. per liter). Add 2 g. of potassium iodide dissolved in 20 ml. of water, stopper, and allow the solution to stand for approximately 5 min. Titrate the liberated iodine with sodium thiosulfate at room temperature until the reddish-brown iodine color becomes quite faint. Then add 5 ml. of freshly prepared starch solution and continue the titration until the final color change becomes pale green with no blue tinge. This final titration should be carried out by twirling the flask at least three times after each addition of the sodium thiosulfate, being sure that there is no further color change, especially at the final stage of the titration. The green end point is definite and sharp. The percentage of chromic acid may be calculated from the following values:

1 ml. of *N* sodium thiosulfate = 0.03334 g. of CrO_3.

Report the amount calculated as percentage of CrO_3.

Zinc

7. (*a*) *Special Solution Required.*—Potassium ferrocyanide solution standardized as follows: Dissolve 42.5 g. of $K_4Fe(CN)_6$ per liter of solution. Dissolve 0.4 g. of pure zinc in 25 ml. of HCl (sp. gr. 1.19). After the zinc has completely dissolved, dilute to 300 ml. Neutralize the solution with NH_4OH, add 2 g. of NH_4Cl and 3 ml. of HCl (sp. gr. 1.19). Titrate the solution with $K_4Fe(CN)_6$ using uranyl acetate as an external indicator on a spot tile and ferrous ammonium sulfate as an

[4] Refer to Blair, "Chemical Analysis of Iron," Fig. 63, p. 138.

internal indicator. Calculate the strength of the $K_4Fe(CN)_6$ solution in terms of grams of zinc as follows:

$$1 \text{ ml. of } K_4Fe(CN)_6 = \frac{\text{grams of zinc}}{\text{ml. of } K_4Fe(CN)_6}$$

(*b*) *Procedure.*—Nearly neutralize 100 ml. of the solution of the sample (Section 5) with NH_4OH, add methyl orange indicator and continue neutralization with weak NH_4OH (1:10) until exactly neutral. There should be no precipitate formed. The solution must be barely acid before beginning the precipitation and the acidity (due to liberated H_2SO_4) should not increase to more than 0.1 *N*. The precipitation of zinc sulfide ceases if the acidity increases to more than 550 mg. of H_2SO_4 per 100 ml. of solution; below 0.1 *N* acid concentration, the precipitation is complete. Pass H_2S through the solution at a rapid rate (at least eight bubbles per second) for at least 40 min., keeping the temperature below 50 C. Filter the solution and wash the precipitate with water.

Dissolve the ZnS precipitate in hot diluted HCl (1:3). Wash the filter paper well with hot water. Boil the H_2S out of the ZnS solution, neutralize with NH_4OH and adjust the volume to 300 ml. Add 2 g. of NH_4Cl, and 3 ml. of HCl (sp. gr. 1.19), and heat to boiling. Titrate the hot solution with standard $K_4Fe(CN)_6$ solution using uranyl acetate as an external indicator on a spot plate, and 1 to 2 drops of ferrous ammonium sulfate as an internal indicator. The percentage of zinc in the specimen may be calculated from the following formula:

$$\text{Zn, per cent} = \frac{\text{ml. of } K_4Fe(CN)_6 \times \text{grams of Zn per ml.}}{\text{wt. of sample}} \times 100$$

$$\text{ZnO, per cent} = \text{percentage of Zn} \times 1.2447$$

Report the amount calculated as percentage of ZnO.

Total Alkali Metals

8. Dissolve 1 g. of the sample in 10 ml. of acetic acid (1:1), add 25 ml. of water and heat until completely dissolved. Dilute to approximately 250 ml. and heat the solution to boiling. Add 20 ml. of lead acetate (10 per cent) and allow to settle. Filter off the precipitate and wash well with hot water. Saturate the filtrate with H_2S, observing the precautions as outlined in the procedure for determining zinc (Section 7 (*b*)); zinc and excess lead will be precipitated. (It is necessary to keep down the concentration of acetic acid to ensure complete removal of the zinc.) Filter, and wash the precipitate with cold water. Boil the filtrate to expel H_2S, then add 3 ml. of H_2SO_4 (1:1), and reduce to a small volume by boiling. Transfer the concentrate to a weighed silica dish and evaporate to dryness on a hot plate. Heat to complete dryness over a bunsen burner. Add solid $(NH_4)_2CO_3$ to the residue and heat till volatilized. Repeat the operation four times, cool the dish, and weigh. Add a few crystals of $(NH_4)_2CO_3$, and again heat the residue until volatilized. Cool, and weigh the dish, and repeat the operation until constant weight is obtained.

The percentage of total alkali may be calculated as follows:

$$\text{Total sulfate } (Na_2SO_4 \text{ and } K_2SO_4), \text{ per cent} = \frac{\text{wt. of alkaline sulfates}}{\text{wt. of sample}} \times 100$$

$$\text{Total alkali } (K_2O), \text{ per cent} = \text{percentage of } K_2SO_4 \times 0.541$$

Report the amount calculated as percentage of K_2O.

NOTE.—The above determination would include both potassium and sodium if present. Flame test for sodium should be made at this point, and if present the procedure for sodium should be followed (see Section 11 (*a*) and (*b*)).

Sulfates

9. Dissolve 1 g. of the sample in 15 ml. of HCl (sp. gr. 1.19). Heat the solution to boiling and add 5 ml. of alcohol by drops to insure the reduction of chromium. Reduce the solution to a small volume by boiling. Dilute the concentrate to 300 ml. with water and neutralize with Na_2CO_3 adding 3.5 g. in excess. Heat the solution to boiling and boil to ensure the conversion of the insoluble sulfates to the soluble form. Allow the precipitate in the solution to settle on a hot plate, and filter. Wash the precipitate with hot water. Neutralize the filtrate with HCl, and boil to expel the CO_2. Adjust the acidity to 2.5 per cent by volume with HCl and add drop by drop 15 ml. of $BaCl_2$ (20 per cent) to the solution, keeping the latter boiling. Allow the precipitate to settle on a hot plate, filter through a weighed Gooch crucible, wash, dry, ignite, cool, and weigh. The increase in weight represents the $BaSO_4$.

$$\text{Sulfate, per cent} = \frac{\text{wt. of } BaSO_4 \times 0.343}{\text{wt. of sample}} \times 100$$

Report the amount calculated as percentage of SO_3.

Chlorine

10. Dissolve 1 g. of the sample in 20 ml. of H_2SO_4 (2:3) and heat the solution to boiling. Add drop by drop 5 ml. of alcohol to the boiling solution; reduction of the chromium is complete when a clear green solution is obtained. Dilute the solution to approximately 250 ml., neutralize with Na_2CO_3 and add a slight excess. Boil the solution a few minutes, filter, and wash. Acidify with diluted HNO_3 (1:1), adding 25 ml. in excess. Add 10 ml. of standard $AgNO_3$ solution (0.05 *N*) and titrate the excess silver nitrate with standard potassium sulfocyanate (0.05 *N*), using ferric sulfate as an internal indicator. A red tinge in the solution indicates the end point. The percentage of chlorine in the sample may be calculated as follows:

$$\text{Chlorine, per cent} = \frac{\text{ml. of } AgNO_3 - (\text{ml. of KCNS} \times \text{factor}) \times \text{grams of Cl per ml. of } AgNO_3}{\text{wt. of sample}} \times 100$$

Report the amount calculated as percentage of chlorine.

Sodium

11. (*a*) *Special Solutions Required.*—Double acetate solution, prepared as follows:

Solution A	
Uranyl acetate	10 g.
Acetic acid (30 per cent)	6 g.
Water	65 g.
Solution B	
Zinc acetate	30 g.
Acetic acid (30 per cent)	3 g.
Water	65 g.

When the above solutions A and B are each completely dissolved, mix them, and allow to cool to 20 ± 1 C. Keep the resulting solution at this temperature for several hours. Filter off the triple salt which crystallizes out and which may be assumed to be saturated with respect to sodium at 20 C. since the reagent usually contains sufficient sodium to give a precipitate.

(*b*) *Procedure.*—Proceed as in the determination of total alkali (Section 8) to the weighed sulfates. Dissolve the sulfates in 10 to 15 ml. of water. Add to the solution 100 ml. of zinc-uranyl acetate reagent and allow to stand at 20 ± 1 C. for 30 to 45 min., while stirring occasionally. (Scratch the side of the beaker occasionally to start crystallization.) Filter the solution through a tared Gooch crucible and wash with five successive amounts of 2 ml. of zinc-uranyl acetate reagent, then with five 2-ml. portions of alcohol (previously

saturated with sodium zinc-uranyl acetate) and finally with ether. Dry at 40 C. for 10 to 15 min., and weigh. The percentage of sodium may be calculated as follows:

Sodium, per cent

$$= \frac{\text{wt. of precipitate} \times 0.01495}{\text{wt. of sample}} \times 100$$

NOTE.—The precipitate is slightly soluble in water, insoluble in the precipitant, alcohol, and ether. Barium, calcium, magnesium, and zinc do not interfere with the precipitation. Potassium has little effect if less than 0.05 g. is present; lithium, phosphates, oxalates, and tartrates should be absent.

Calcium

12. Proceed as in the determination of sulfates (Section 9) to the acidification of the sodium carbonate solution. Make the solution ammoniacal and then slightly acid with acetic acid. Add 15 ml. of saturated ammonium acetate solution and again make ammoniacal with NH_4OH and allow to stand on a hot plate over night. Filter off any precipitate in the morning. Wash free from oxalates with NH_4OH (10 per cent). Dissolve the oxalate precipitate in hot diluted H_2SO_4. Dilute the solution to approximately 300 ml. and add 10 ml. of H_2SO_4. Heat the solution to boiling and titrate with $KMnO_4$ solution (0.1 *N*).

Calcium, per cent

$$= \frac{\text{ml. of } KMnO_4 \times N \times 0.20}{\text{wt. of sample}} \times 100$$

where N = normality of the $KMnO_4$ solution.

Ammonia[5]

13. Distill 1 g. of the sample with NaOH solution, expelling the ammonia and collecting it in a known excess of H_2SO_4 (0.1 *N*). Titrate the excess H_2SO_4 with NaOH (0.1 *N*).

[5] This procedure is occasionally but not usually employed for the analysis of zinc yellow pigments.

Standard Specifications for

RAW TUNG OIL[1]

A.S.T.M. Designation: D 12 – 41

ADOPTED, 1933; REVISED, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 12; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover raw tung oil.

Properties

2. Raw tung oil shall conform to the following requirements:

Specific gravity, 15.5/15.5 C...	0.940[a] to 0.943
Acid number (alcohol-benzol),[b] max.....	8.0
Saponification number.....	190 to 195
Unsaponifiable matter, max., per cent.....	0.75
Iodine number (Wijs), min.	163
Appearance.....	clear and transparent at 65 C.
Color[b].....	not darker than a freshly prepared solution of 1.0 g. of $K_2Cr_2O_7$ in 100 ml. of pure H_2SO_4 (sp. gr. 1.84), or its equivalent in iron-cobalt solution or in Lovibond glasses.
Heating test, max., minutes...	12
Refractive index at 25 C.....	1.5165 to 1.520

[a] For American grown tung oil, the minimum specific gravity may be as low as 0.938.

[b] If tung oil of low acid number and pale color is desired, the maximum acid number should be reduced to 3.0, and the maximum color to 0.03 g. of $K_2Cr_2O_7$ per 100 ml. of H_2SO_4 (or iron-cobalt or Lovibond equivalents).

Sampling

3. The method of sampling described in Paragraph (*a*) shall be used whenever it is feasible to apply it. To meet conditions when this procedure is not applicable, the method of sampling described in Paragraph (*b*) or (*c*) shall be used, according to the special conditions that obtain.

(*a*) *During Loading of Tank Cars or Filling of Containers for Shipment.*—The purchaser's inspector shall draw a sample at the discharge pipe where it enters the receiving vessel or vessels. The total sample shall be not less than 1 gal. per tank car or 10,000 gal. equivalent, and shall be a composite of small samples of not more than 1 pt. each taken at regular intervals during the entire period of loading or filling.

The sample thus obtained shall be thoroughly mixed, and from this composite sample three portions of not less than 1 qt. each shall be placed in clean, dry glass bottles or tin cans, which shall be filled with the sample and securely stoppered with new clean corks or well-fitting metal covers or caps. These shall

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to their present adoption as standard, these specifications were adopted in 1915, revised in 1916, but withdrawn and replaced by D 12 – 23 T which was published as tentative from 1922 to 1933, being revised in 1923 and 1925.

be sealed and labeled distinctly by the inspector, and one delivered to the purchaser, one to the seller, and the other held for check in case of dispute.

(*b*) *From Loaded Tank Cars or Other Large Vessels.*—The total sample shall be not less than 1 gal. per tank car or 10,000 gal. equivalent, and shall be a composite of numerous small samples of not more than 1 pt. each, taken from the top, bottom, and intermediate points by means of a glass or metal container with removable stopper or top. This device, attached to a suitable pole, shall be lowered to various desired depths, when the stopper or top shall be removed and the container allowed to fill. The sample thus obtained shall be handled as described in Paragraph (*a*). For large shipments in freighters ranging from 400 to 1000 tons, the samples shall first be drawn from the top and bottom of both port and starboard sections of the tank. These samples shall be visually examined, and if the general appearance gives indications that the oil is satisfactory, pumping shall be started and 1-pt. samples drawn from a bleeder in the discharge line at least once in every 30 min., so that the total mixed sample amounts to as many gallons as there are units of 10,000 gal. in the cargo. These individual samples shall be composited to make one uniform sample representative of the entire cargo, and this shall be handled as described in Paragraph (*a*).

(*c*) *From Barrels and Drums.*—Not less than 10 per cent of the packages in any shipment or delivery of barrels and drums shall be sampled. The packages shall be shaken, rolled, and stirred to thoroughly mix the contents. The samples from the individual containers shall be taken through the bung hole, or holes not less than 1¼ in. in diameter bored in the head or side for the purpose. The apparatus for drawing the sample shall consist of a glass tube (or "thief") about 1 in. in diameter and somewhat longer than the length or diameter of the oil container, and a conical stopper that will fit the glass tube and is not more than ½ in. in length, fastened to a stiff metal rod not more than ¼ in. in diameter and not less than 4 in. longer than the glass tube. The stopper shall be lowered by the rod until it rests on the bottom of the container, the tube slipped down slowly over the rod, and finally pressed on the stopper. By holding the tube and the rod, the column of oil can then be removed. This process shall be repeated until the required amount of sample is obtained, which shall be not less than 1 gal. This shall be mixed and handled as described in Paragraph (*a*). During the winter, when the oil is very often in a solid condition, the same procedure shall be followed, replacing the thief with a tallow trier, which is essentially a graduated piece of steel tubing semicircular in cross-section and about 3 ft. in length. This shall be pushed into the oil, turned a few times, and withdrawn, thereby removing a solid core of oil.

Methods of Testing

4. The properties enumerated in these specifications shall be determined in accordance with the Standard Methods of Testing Drying Oils (A.S.T.M. Designation: D 555) of the American Society for Testing Materials.[3]

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for

RAW SOYBEAN OIL[1]

A.S.T.M. Designation: D 124 – 41

ADOPTED, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 124; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover raw soybean oil.

Properties

2. Raw soybean oil shall conform to the following requirements:

Specific gravity, 15.5/15.5 C., min.	0.924
Acid number, max.	3.0
Saponification number, min.	190
Unsaponifiable matter, max., per cent	1.5
Iodine number (Wijs), min.	131
Loss on heating, 105 to 110 C., max., per cent	0.3
Appearance	clear and transparent at 65 C.
Color	not darker than a freshly prepared solution of 1.0 g. of $K_2Cr_2O_7$ in 100 ml. of pure H_2SO_4 (sp. gr. 1.84), or its equivalent in iron-cobalt solution or in Lovibond glasses.
Foots, max., per cent	2.5

Methods of Testing

3. The properties enumerated in these specifications shall be determined in accordance with the Standard Methods of Testing Drying Oils (A.S.T.M. Designation: D 555) of the American Society for Testing Materials.[3]

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1922 to 1941, being revised in 1933, 1937, and 1941.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for

PERILLA OIL, RAW OR REFINED[1]

A.S.T.M. Designation: D 125 – 41

ADOPTED, 1923; REVISED, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 125; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover raw or refined perilla oil.

Properties

2. Perilla oil, raw or refined, shall conform to the following requirements.

Specific gravity, 15.5/15.5 C., min.	0.932
Acid number, max.	5.0
Saponification number, min.	190
Unsaponifiable matter, max., per cent	1.5
Iodine number (Hanus), min.	191
Loss on heating at 105 to 110 C., max., per cent	0.2
Appearance	clear and transparent at 65 C.
Color	not darker than a freshly prepared solution of 1.0 g. of $K_2Cr_2O_7$ in 100 ml. of pure H_2SO_4 (sp. gr. 1.84), or its equivalent in iron-cobalt solution or in Lovibond glasses.
Foots, max., per cent	2.5

Methods of Testing

3. The properties enumerated in these specifications shall be determined in accordance with the Standard Methods of Testing Drying Oils (A.S.T.M. Designation: D 555) of the American Society for Testing Materials.[3]

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1922 to 1923.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

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Standard Specifications for
RAW LINSEED OIL[1]

A.S.T.M. Designation: D 234 – 28

ADOPTED, 1927; REVISED, 1928.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 234; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover raw linseed oil.

Properties

2. Linseed oil shall be the pure oil pressed from flaxseed and shall conform to the following requirements:

Specific gravity, 15.5/15.5 C.	0.931 to 0.936
Acid number, max.	4.0
Saponification number	189.0 to 195.0
Unsaponifiable matter, max., per cent	1.50
Iodine number (Wijs),[a] min.	177.0
Loss on heating at 105 to 110 C., max., per cent	0.2
Color	not darker than a freshly prepared solution of 1.0 g. of $K_2Cr_2O_7$ in 100 ml. of pure H_2SO_4 (sp. gr. 1.84).
Foots, max., per cent:	
Heated oil	1.0
Chilled oil	4.0

[a] If linseed oil of the high iodine number type is desired, the minimum iodine number, as specified above, should be changed to 188.0.

Methods of Testing

3. The properties enumerated in these specifications shall be determined in accordance with the Standard Methods of Testing Drying Oils (A.S.T.M. Designation: D 555) of the American Society for Testing Materials.[3]

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1926 to 1927, being revised in 1927. Editorially revised and rearranged in 1939.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

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Standard Specifications for

BOILED LINSEED OIL[1]

A.S.T.M. Designation: D 260 – 33

ADOPTED, 1933.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 260; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover boiled linseed oil.

Properties

2. Boiled linseed oil shall be pure linseed oil that has been treated by heating and incorporating compounds of lead, and at the option of the manufacturer suitable compounds of other drying metals, so as to produce a product that will dry rapidly. It shall be clear, free from sediment, and shall conform to the following requirements:

Specific gravity, 15.5/15.5 C	0.931 to 0.945[a]
Acid number, max.	7.5
Saponification number	189.0 to 195.0
Unsaponifiable matter, max., per cent	1.50
Iodine number (Wijs), min.	170.0
Loss on heating at 105 to 110 C., max., per cent	0.2
Time of drying on glass, max., hr.	18
Ash, max., per cent	0.50
Lead, min., per cent	0.05

[a] When a high viscosity type of boiled linseed oil is required, the specific gravity shall be not less than 0.937.

Methods of Testing

3. The properties enumerated in these specifications shall be determined in accordance with the Standard Methods of Testing Drying Oils (A.S.T.M. Designation: D 555) of the American Society for Testing Materials.[3]

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1927 to 1933, being revised in 1928. Editorially revised and rearranged in 1939.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

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Standard Specifications for

SPIRITS OF TURPENTINE[1]



A.S.T.M. Designation: D 13 – 34

ADOPTED, 1934.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 13; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover the following kinds of spirits of turpentine: Gum spirits of turpentine, steam-distilled wood turpentine, sulfate wood turpentine, and destructively-distilled wood turpentine.

The purchaser should specify the kind of spirits of turpentine desired.

Properties

2. Spirits of turpentine shall be pure and shall conform to the requirements prescribed in Table I.

TABLE I.—PHYSICAL REQUIREMENTS.

	Gum Spirits of Turpentine		Wood Turpentine					
			Steam Distilled		Sulfate		Destructively Distilled	
	Maximum	Minimum	Maximum	Minimum	Maximum	Minimum	Maximum	Minimum
Specific gravity, 15.5/15.5 C	0.875	0.860	0.875	0.860	0.875	0.860	0.866	0.860
Refractive index at 20 C., *D* line	1.478	1.465	1.478	1.465	1.478	1.465	1.483	1.463
Residue after polymerization with 38 *N* H_2SO_4:								
Volume, per cent	2	.....	2	.....	2	.....	2	.....
Refractive index at 20 C	.....	1.500	.....	1.500	.....	1.500	.....	1.480
Initial boiling point at 760-mm. pressure, deg. Cent.	160	150	160	150	160	150	157	150
Distilling below 170 C. at 760-mm. pressure, per cent	.....	90	.....	90	.....	90	.....	60
Distilling below 180 C. at 760-mm. pressure, per cent	.....	.....	.....	.....	.....	.....	.....	90

Appearance

3. Spirits of turpentine shall be clear and free from suspended matter and water.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to their present adoption as standard, these specifications were published as tentative from 1914 to 1915, being revised in 1915. They were adopted in 1915, published as standard from 1915 to 1921, withdrawn and republished as tentative from 1920 to 1924, adopted as standard in 1924, published as standard from 1924 to 1933, being revised in 1926. They were withdrawn and republished as tentative from 1933 to 1934 to replace the former Standard Specifications D 13 – 26 and D 236 – 27.

Color

4. The color shall be "standard" or better.

Odor

5. The odor shall be mild and characteristic of the kind of spirits of turpentine specified. If desired, deliveries shall conform to the odor of the sample agreed upon by the purchaser and the seller.

Methods of Testing

6. The material shall be sampled and the properties enumerated in these specifications determined in accordance with the Tentative Methods of Sampling and Testing Turpentine (A.S.T.M. Designation: D 233) of the American Society for Testing Materials.[3]

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

AMERICAN ASSOCIATION STATE
HIGHWAY OFFICIALS STANDARD
A.A.S.H.O. NO.: M 128-42

Standard Specifications for
PETROLEUM SPIRITS (MINERAL SPIRITS)[1]

A.S.T.M. Designation: D 235 – 39

ADOPTED, 1939.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 235; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications apply only to petroleum distillates.

Properties

2. The material shall conform to the following requirements:

Appearance	clear and free of suspended matter and water
Color	water white, not darker than No. 21 Saybolt Chromometer
Flash point, min.	100 F. (38 C.)
Blackening	shall not blacken or corrode clean metallic copper in 30 min. at boiling point of spirits
Distillation:	
Percentage recovered at 350 F. (177 C.), min.	50
End point	410 F. (210 C.)
Acidity of distillation residue	neutral

Methods of Testing

3. The properties enumerated in these specifications shall be determined in accordance with the following methods of test:

(*a*) *Color.*—Standard Method of Test for Color of Refined Petroleum Oil by Means of Saybolt Chromometer (A.S.T.M. Designation: D 156) of the American Society for Testing Materials.[3] A fresh aqueous solution of potassium dichromate in distilled water, containing 0.0048 g. $K_2Cr_2O_7$ per liter, is approximately equivalent to color No. 21 Saybolt Chromometer.

(*b*) *Flash Point.*—Standard Method of Test for Flash Point by Means of the Tag Closed Tester (A.S.T.M. Designation: D 56) of the American Society for Testing Materials.[3]

(*c*) *Blackening.*—A clean strip of mechanically polished pure sheet copper, about $\frac{1}{2}$ in. (1.3 cm.) in width and 3 in (7.5 cm.) in length, shall be placed in a glass test tube about $\frac{3}{4}$ in. (1.9 cm.) in width and 18 in. (46 cm.) in length. Sufficient of the sample to be tested shall be added to completely cover the strip and heated rapidly to boiling. It is most convenient to heat the tube by immersion in an oil bath maintained at a

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1926 to 1939, being revised in 1934.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

temperature slightly higher than the initial boiling point of the sample. The sample shall be kept boiling for 30 min. without any actual distillation taking place and the copper strip then examined for blackening. A slight tarnish shall be disregarded, but any marked blackening shall be cause for rejection.

(*d*) *Distillation.*—Standard Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (A.S.T.M. Designation: D 86) of the American Society for Testing Materials.[3]

(*e*) *Acidity.*—The cooled residue from the distillation flask shall be collected in a test tube, three volumes of distilled water added, and the contents shaken thoroughly. The mixture shall be allowed to separate and the aqueous layer removed to a clean test tube by means of a pipette. One drop of a 1 per cent solution of methyl orange shall be added. No pink or red color should be formed.

Standard Methods of
TESTING DRYING OILS[1]

A.S.T.M. Designation: D 555 – 41

ADOPTED, 1939; REVISED, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 555; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods cover the test procedures to be applied to drying oils for use in paints and varnishes.

Reagents

2. (*a*) *Standard Sodium Hydroxide Solution.*—Prepare a stock concentrated solution of sodium hydroxide by dissolving NaOH in water in the proportion of 200 g. of NaOH to 200 ml. of water. Allow this solution to cool and settle in a stoppered bottle for several days. Decant the clear liquid from the precipitate of Na_2CO_3 into another clean bottle. Add clear barium hydroxide solution until no further precipitate forms. Again allow to settle until clear. Draw off about 175 ml. and dilute to 10 liters with freshly boiled distilled water. Preserve in a stock bottle provided with a large guard tube filled with soda lime. Determine the exact strength by titrating against pure benzoic acid (C_6H_5COOH) using phenolphthalein as indicator.[3] This solution will be approximately 0.25 *N*, but do not attempt to adjust it to any exact value. Determine its exact strength and make proper corrections in using it.

(*b*) *Alcoholic Sodium Hydroxide Solution.*—Dissolve pure NaOH in 95 per cent ethyl alcohol in the proportion of about 22 g. per 1000 ml. Let stand in a stoppered bottle. Decant the clear liquid into another bottle, and keep well stoppered. This solution should be colorless or only slightly yellow when used; it will keep colorless longer if the alcohol is previously treated with NaOH (about 80 g. to 1000 ml.), kept at about 50 C. for 15 days, and then distilled. For an alternate method see *Journal*, American Chemical Society, 1906, p. 395.

(*c*) *Sulfuric Acid Solution (0.5 N).*—Add about 15 ml. of H_2SO_4 (sp. gr. 1.84) to distilled water, cool, and dilute to 1000 ml. Determine the exact strength by titrating against freshly standardized

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] These methods were editorially rearranged and issued in 1939 under the designation D 555. They formerly constituted a portion of the Standard Specifications for Raw Tung Oil (D 12 – 33), Perilla Oil, Raw or Refined (D 125 – 33), Raw Linseed Oil (D 234 – 28), Boiled Linseed Oil (D 260 – 33), and Tentative Specifications for Raw Soybean Oil (D 124 – 37 T).

[3] National Bureau of Standards *Scientific Paper No. 183.*

NaOH or by any other accurate method. Either adjust to exactly 0.5 *N* strength or leave as originally made, applying appropriate correction.

(*d*) *Wijs Iodine Monochloride Solution.*—Dissolve iodine in glacial acetic acid having a melting point of 14.7 to 15 C. and free from reducing impurities, in such proportion that 13 g. of iodine will be present in 1000 ml. of solution. The preparation of the iodine monochloride solution presents no great difficulty, but it shall be done with care and accuracy in order to obtain satisfactory results. There shall be in the solution no sensible excess either of iodine or more particularly of chlorine over that required to form the monochloride. This condition is most satisfactorily attained by dissolving in the whole of the acetic acid to be used the requisite quantity of iodine, using a gentle heat to assist the solution if it is found necessary. The halogen content can be accurately measured by titration of a portion of the solution with 0.1 *N* sodium thiosulfate, using the potassium iodide solution (15 per cent) and the starch indicator as described in Section 9 in the procedure for the determination of the iodine number. Set aside a small portion of this solution while pure, and pass dry chlorine into the remainder until the halogen content of the whole solution is doubled. Ordinarily, it will be found that by passing the chlorine into the main part of the solution until the characteristic color of free iodine has just been discharged, there will be a slight excess of chlorine, which is corrected by the addition of the requisite amount of the unchlorinated portion until all free chlorine has been destroyed. A slight excess of iodine does little or no harm, but excess of chlorine must be avoided.

(*e*) *Hanus Solution.*—Dissolve 13.2 g. of iodine in 1000 ml. of glacial acetic acid (99.5 per cent) that will not reduce chromic acid. Add enough bromine to double the halogen content, determined by titration (3 ml. of bromine is about the proper amount). The iodine may be dissolved by the aid of heat, but the solution should be cold when the bromine is added.

(*f*) *Chloroform, U.S.P.*

(*g*) *Starch Solution.*—Stir up 2 to 3 g. of potato starch or 5 g. of soluble starch with 100 ml. of 1 per cent salicylic acid solution, add 300 to 400 ml. of boiling water, and boil the mixture until the starch is practically dissolved. Dilute to 1 liter.

(*h*) *Potassium Iodide Solution (15 per cent).*—Dissolve 150 g. of KI, free of KIO_3, in distilled water and dilute to 1000 ml.

(*i*) *Standard Sodium Thiosulfate Solution.*—Dissolve pure sodium thiosulfate in distilled water that has been well boiled to free it from CO_2, in such proportion that 24.83 g. of $Na_2S_2O_3 \cdot 5H_2O$ will be present in 1000 ml. of the solution. It is best to let this solution stand for about two weeks before standardizing. Standardize with pure resublimed iodine.[4] This solution will be approximately 0.1 *N*, and it is best to leave it as it is after determining its exact iodine value, rather than to attempt to adjust it to exactly 0.1 *N* strength. Preserve in a stock bottle with a guard tube filled with soda lime.

(*j*) *Acid Calcium Chloride Solution.*—Saturate with $CaCl_2$ a mixture of 90 parts of water and 10 parts of HCl (sp. gr. 1.19).

(*k*) *Acetone.*—Acetone shall conform to the Standard Specifications for Acetone (A.S.T.M. Designation: D 329) of the American Society for Testing Materials.[5]

[4] Treadwell-Hall, *Analytical Chemistry*, Vol. II, Sixth edition, pp. 551 and 553.

[5] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Preparation of Sample

3. Thoroughly mix the laboratory sample by shaking, stirring, or pouring it from one vessel to another. Take the samples for the individual tests from this thoroughly mixed laboratory sample.

Specific Gravity

4. Use a pycnometer, accurately standardized and having a capacity of at least 25 ml., or any other equally accurate method, making the test at 15.5 C., the specific gravity of water being 1.000 at 15.5 C.

Acid Number of Linseed Oil and Raw Tung Oil

5. To determine the acid number of boiled or raw linseed oil and of raw tung oil, weigh from 5 to 10 g. of the sample and transfer to a 300-ml. Erlenmeyer flask. Add 50 ml. of a mixture of equal parts by volume of 95 per cent ethyl alcohol and c.p. reagent benzol. (This mixture should be previously titrated to a very faint pink with diluted alkali solution, using phenolphthalein as an indicator.) Add phenolphthalein indicator, and titrate at once to a faint permanent pink color with 0.2 *N* NaOH or KOH solution. Calculate the acid number (milligrams of KOH required for 1 g. of the oil).

Acid Number of Perilla Oil and Raw Soybean Oil

6. To determine the acid number of raw or refined perilla oil and of raw soybean oil, weigh from 5 to 10 g. of the oil and transfer to a 300-ml. Erlenmeyer flask. Add 50 ml. of neutral 95 per cent ethyl alcohol. Put a condenser loop inside the neck of the flask. Heat on a steam bath for 30 min. Cool, and add phenolphthalein indicator. Titrate to a faint permanent pink color with the 0.2 *N* NaOH or KOH solution. Calculate the acid number (milligrams of KOH required for 1 g. of the oil).

Saponification Number

7. Weigh about 2 g. of the sample in a 300 ml. Erlenmeyer flask. Add 25 ml. of alcoholic NaOH or KOH solution. Put a condenser loop inside the neck of the flask and heat on the steam bath for 1 hr. Cool, add phenolphthalein as indicator, and titrate with 0.5 *N* H_2SO_4. Run two blanks with the alcoholic NaOH or KOH solution. These should check within 0.1 ml. of 0.5 *N* H_2SO_4. From the difference between the number of milliliters of 0.5 *N* H_2SO_4 required for the blank and for the determination, calculate the saponification number (milligrams of KOH required for 1 g. of the oil).

Unsaponifiable Matter

8. (*a*) Weigh 8 to 10 g. of the sample. Transfer to a 250-ml. long-neck flask. Add 5 ml. of NaOH solution (1:1) and 50 ml. of 95 per cent ethyl alcohol. Put a condenser loop inside the neck of the flask and boil for 2 hr. Occasionally agitate the flask to break up the liquid, but do not project the liquid onto the sides of the flask. At the end of 2 hr., remove the condenser and allow the liquid to boil down to about 25 ml.

(*b*) Transfer to a 500-ml. glass-stoppered separatory funnel, rinsing with water. Dilute with water to 250 ml., and add 100 ml. of redistilled ether. Stopper and shake for 1 min. Let stand until the separation between the two layers is sharp and clear. Draw off all but one or two drops of the aqueous layer into a second 500-ml. separatory funnel and repeat the process, using 60 ml. of ether. After thorough separation, draw off the aqueous solution into a 400-ml. beaker, then transfer the ether solution to the first separatory funnel, rinsing down with a little water. Return

the aqueous solution to the second separatory funnel and extract again with 60 ml. of ether in a similar manner, finally drawing the aqueous solution into the beaker and rinsing the ether into the first separatory funnel.

(*c*) Shake the combined ether solution with the combined water rinsings and let stand until the separation between the two layers is sharp and clear. Draw off the water and add it to the main aqueous solution. Shake the ether solution with two portions of water (about 25 ml. each). Add these to the main water solution.

(*d*) Swirl the separatory funnel so as to bring the last drops of water down to the stopcock, and draw off until the ether solution just fills the bore of the stopcock. Wipe out the stem of the separatory funnel with a bit of cotton on a wire. Draw off the ether solution (portionwise if necessary) into a 250-ml. flask and distill. While still hot, drain the flask into a small weighed beaker, rinsing with a little ether. Evaporate this ether, cool the beaker, and weigh.

NOTE.—The unsaponifiable oil from adulterated drying oils may be volatile and as a consequence may evaporate on long heating. Therefore, heat the beaker on a warm plate, occasionally blowing out with a current of dry air. Discontinue heating as soon as the odor of ether is gone.

Iodine Number, Wijs

9. Place a small quantity of the sample in a small weighing burette or beaker. Weigh accurately. Transfer, by dropping, from 0.09 to 0.15 g. of oil (0.16 to 0.19 g. for tung oil) to a 500-ml. bottle having a well-ground glass stopper, or an Erlenmeyer flask having a specially flanged neck for the iodine test. Reweigh the burette or beaker and determine the amount of sample used. Add 10 ml. of chloroform. Whirl the bottle to dissolve the sample. Add 10 ml. of chloroform to each of two empty bottles like that used for the sample. Add to each bottle 25 ml. of the Wijs solution and let stand with occasional shaking for 1 hr. in a dark place at a temperature from 21 to 23 C. Add 10 ml. of the potassium iodide solution (15 per cent) and 100 ml. of water. Titrate with 0.1 *N* sodium thiosulfate, using starch as an indicator. The titrations on the two blank tests should agree within 0.1 ml. From the difference between the average of the blank titrations and the titration on the sample and the iodine value of the thiosulfate solution, calculate the iodine number of the sample tested. Report the iodine number in centigrams of iodine for 1 g. of the oil.[6]

Iodine Number, Hanus

10. Place a small quantity of the sample in a small weighing burette or beaker. Weigh accurately. Transfer, by dropping, about 0.15 g. (0.10 to 0.20 g.) into a 500-ml. bottle having a well-ground glass stopper, or an Erlenmeyer flask having a specially flanged neck for the iodine test. Reweigh the burette or beaker and determine the amount of sample used. Add 10 ml. of chloroform. Whirl the bottle to dissolve the sample. Add 10 ml. of chloroform to each of two empty bottles like that used for the sample. Add to each bottle 25 ml. of the Hanus solution and let stand with occasional shaking for 30 min. Add 10 ml. of the potassium iodide solution (15 per cent) and 100 ml. of water, and titrate with standard sodium thiosulfate using starch as indicator. The titrations on the two blank tests should agree within 0.1 ml. From the difference between the average of the blank titrations and the titration on the sample

[6] In determining the iodine value of tung oil it is always well to include a test on a sample of tung oil of known iodine value. This may be kept in a dark-colored bottle as a reference standard.

and the iodine value of the thiosulfate solution, calculate the iodine number of the sample tested. Report the iodine number in centigrams of iodine for 1 g. of the oil.

Loss on Heating

11. Place 10 g. of the sample in an accurately weighed 200-ml. Erlenmeyer flask (for linseed oil use a 50-ml. flask), and weigh. Heat in an oven at a temperature between 105 and 110 C. for 30 min., then cool and weigh. Calculate the percentage loss. This determination shall be made in a current of CO_2 gas.

Appearance

12. (*a*) Transfer a portion of the sample to a clear glass tube and note the appearance.

(*b*) In the case of perilla and raw tung oils, if the oil is cloudy when cold, it should become clear and transparent when heated at 65 C. for 5 min.

Color

13. (*a*) Prepare a fresh solution of 1 g. of pure $K_2Cr_2O_7$ in 100 ml. of pure colorless H_2SO_4 (sp. gr. 1.84). Place the oil and colored solution in separate thin-walled clear-glass tubes of the same diameter (1 to 2 cm.) to a depth of not less than 2.5 cm., and compare the depths of color by looking transversely through the columns of liquid by transmitted light.

(*b*) *Iron-Cobalt Liquid Equivalent.*—The iron-cobalt liquid equivalent to the above $K_2Cr_2O_7$ solution may be prepared as follows: Mix 29.4 ml. of solution No. 1 with 17.6 ml. of solution No. 2 and 53.0 ml. of solution No. 3. The three base solutions referred to have the following composition:

Solution No. 1:	
$FeCl_3.6H_2O$	5.0 g.
HCl (2 per cent)	1.2 g.
Solution No. 2:	
$CoCl_2.6H_2O$	1.0 g.
HCl (2 per cent)	3.0 g.
Solution No. 3:	
Aqueous solution of HCl (2 per cent)	

(*c*) *Lovibond Equivalent.*—The Lovibond equivalent to the above iron-cobalt solution shall be as follows: red 17.5, yellow 120.0, and blue 0.20.

The depth of liquid shall be 10.5 mm.

Foots

14. (*a*) With all materials at a temperature between 20 and 27 C., mix by shaking for exactly 1 min. in a graduated tube 25 ml. of the well-shaken sample of oil, 25 ml. of acetone, and 10 ml. of the acid calcium chloride solution. Clamp the tube in a vertical position where settling may take place for 24 hr. The temperature during this period should be between 20 and 27 C.

(*b*) At the end of the 24 hr., determine the volume of the stratum lying between the clear calcium chloride solution and the clear acetone and oil mixture to the nearest 0.1 ml. or fraction thereof. This volume multiplied by four expresses the amount of foots present as a percentage by volume.

(*c*) The graduated tube referred to may be a burette or a color comparison tube. It should have an internal diameter of from 1.0 to 1.5 cm., and a capacity of not less than 70 ml. The graduations in 0.1 ml. should extend at least from 10 to 50 ml. above the bottom of the tube.

Heated Oil Test

15. Heat a portion of the oil to 65 C.; hold it within 2 C. of that temperature for 10 min.; then cool it to room temperature (20 to 27 C.). Subject the

sample promptly to the foots test as described in Section 14.

Chilled Oil Test

16. Heat a portion of the oil to 65 C.; hold it within 2 C. of that temperature for 10 min.; then place it in a dry clean bottle, stopper tightly, and place in a cracked ice and water mixture (0 C.) for exactly 2 hr. At the end of this time, place the bottle for exactly 30 min. in a water bath at 25 C; then subject promptly to the foots test as described in Section 14.

Heating Test

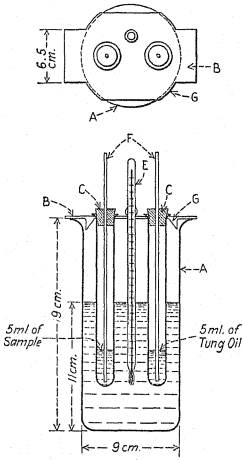
17. (*a*) *Apparatus.*—The apparatus shall consist of the following:

Test Tubes.—Test tubes for the oil, 15 cm. by 16 mm., with a mark near the bottom to indicate 5 ml. Each tube shall be closed by a cork so perforated that a glass rod 3 mm. in diameter may be moved freely. The tubes shall each weigh 14.5 ± 2.0 g. (The purpose of this requirement is to control the wall thickness.)

Thermometer.—An A.S.T.M. Tung Oil Thermometer graduated in Centigrade degrees, having a range of 240 to 310 C., and conforming to the requirements for this thermometer as prescribed in the Standard Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1) of the American Society for Testing Materials.[5]

(*b*) *Procedure.*—Fill a 1000-ml., tall-form glass beaker (19 cm. in height, about 9 cm. in diameter) with cottonseed oil or other suitable oil to a height of 11 cm. Place the thermometer as shown in Fig. 1 so that the bottom will be level with the bottoms of the test tubes. The thermometer may be suspended in the bath by a wire or may be sealed to a glass rod as shown in Fig. 1.

When the bath temperature is 293 C. (560 F.) and rising very slowly at this point, place the tube containing 5 ml. of the oil to be tested and the tube containing 5 ml. of a control sample of known value into the large holes of the cover. Note the time. Remove the source of heat for about 45 sec. and then



A—1000-ml., tall-form beaker, 9 cm. in diameter by 19 cm. in height.
B—support plate about 6.5 cm. in width, made of monel metal, aluminum, or stainless steel.
C—corks.
D—test tubes 15 cm. by 16 mm.
E—thermometer, range 240 to 310 C.
F—glass rods 3 mm. in diameter.
G—guide to prevent cover from slipping.

FIG. 1.—Apparatus For Heating Test of Tung Oil.

reapply. Before 2 min. have elapsed the temperature of the bath will have fallen to 282.2 C. (540 F.), at which point it should be kept as steady as possible. When the samples have been in the bath 9 min., raise the glass rods at intervals of 15 sec. Note the time when each sample becomes firmly set. At this point the oil will be so stiff that

the entire tube may be lifted by aid of the rod. As setting or jellying takes place within a few seconds of fluidity, a good end point is afforded. Remove the samples. Heat the bath again to 293 C. (560 F.), and repeat the experiment with fresh portions of the sample.

NOTE.—No stirrer is used in the bath. A screen around the bath enables the temperature to be reached more easily. When the bath oil has become tarry and viscid, it should be renewed; otherwise heating may be irregular.

Quality Test[7]

18. (*a*) *Apparatus.*—The apparatus shall consist of the following:

Casserole.—An ordinary vitreous enameled iron casserole having a bottom diameter of 7.5 cm. (3 in.).

Tripod.—A wide flange tripod with a 7.5-cm. (3-in.) opening (the object of the flange is to prevent superheating of the sides of the casserole).

Thermometer.—An A.S.T.M. Low Distillation Thermometer graduated in Centigrade degrees, having a range of 0 to 300 C., and conforming to the requirements for this thermometer as prescribed in the Standard Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1) of the American Society for Testing Materials.[6]

(*b*) *Procedure.*—Put into the casserole 150 g. of the oil and heat so that the temperature reaches 282.2 C. (540 F.) in 4 min. ± 30 sec., stirring with the thermometer. Turn down the flame and maintain the temperature as near 282.2 ± 1 C. (540 ± 2 F.) as possible, stirring with the thermometer until, on lifting the thermometer, the oil drops with a pronounced string, showing that polymerization has started. The time required after reaching 282.2 ± 1 C. (540 ± 2 F.) until the string is noted, is the time of the heat test. For pure tung oils this will not exceed 8 min. As soon as the oil strings, remove the lamp and the thermometer, and stir with a stiff spatula until the oil is solid. After stringing, a pure tung oil will require not over 40 sec. more to become solid. When solid, allow to stand exactly 1 min., then turn out, upside down, on clean paper and cut at once with a clean spatula.

Pure tung oil gives a gel that is dry, not adhering to the spatula when cut, and firm, crumbling under pressure of the spatula without sticking; and the cut portions should crumble under pressure like dry bread crumbs. Adulterated tung oil gives a gel that is soft, sticky, and which will not crumble.

Refractive Index

19. Use a properly standardized Abbé refractometer at 25 C., or any other equally accurate instrument.

Time of Drying on Glass

20. Flow the sample over a perfectly clean glass plate. Place the plate in a vertical position in air at 30 ± 2 C. and a relative humidity of 32 ± 4 per cent. After about 2 hr., test the film at intervals with the finger at points not less than 2.5 cm. from the edges. The film shall be considered dry when it no longer adheres to the finger and does not rub up appreciably when the finger is lightly rubbed across the surface.

Ash

21. Weigh a porcelain crucible or dish and add to it from 10 to 25 ml. of the sample, carefully weighing the amount added. Place on a stone slab on the floor of a hood. Ignite by playing the flame of a burner on the surface of the oil and allow to burn quietly until most of the oil is burned off; then transfer to a muffle or over a flame and continue

[7] Based on Worstall Test.

heating at a low temperature (not over a dull red) until all carbonaceous matter is consumed. Cool, weigh, and calculate the percentage of ash.

Lead

22. Dissolve the ash in diluted HNO_3 (1:3) to which a little H_2O_2 has been added, and determine lead by the sulfate or any other equally accurate method.

Matter Insoluble in Chloroform

23. Dissolve 10 g. of the sample in sufficient U.S.P. chloroform to obtain a fluid solution. Filter through a dried and weighed Gooch crucible, wash with chloroform until the oil is removed, and dry at 105 to 110 C. Cool, weigh, and calculate the percentage of matter insoluble in chloroform.

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Standard Method of Test for

DISTILLATION OF GASOLINE, NAPHTHA, KEROSINE, AND SIMILAR PETROLEUM PRODUCTS[1]

A.S.T.M. Designation: D 86 – 40

ADOPTED, 1927; REVISED, 1930, 1935, 1938, 1940.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 86; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test is intended for use in the distillation of gasoline, naphtha, kerosine, and similar petroleum products.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Flask.*—A standard 100-ml. Engler distillation flask, as shown in Fig. 1, conforming to the following dimensions and permissible variations:

	Centimeters	Inches	Permissible Variations	
			Centimeters	Inches
Diameter of bulb, outside	6.5	2.56	±0.2	±0.08
Diameter of neck, inside	1.6	0.63	±0.1	±0.04
Length of neck	15.0	5.91	±0.4	±0.16
Length of vapor tube	10.0	3.94	±0.3	±0.12
Diameter of vapor tube, outside	0.6	0.24	±0.05	±0.02
Diameter of vapor tube, inside	0.4	0.16	±0.05	±0.02
Thickness of vapor tube wall	0.1	0.04	±0.05	±0.02

The position of the vapor tube shall be 9 ± 0.3 cm. (3.55±0.12 in.) above

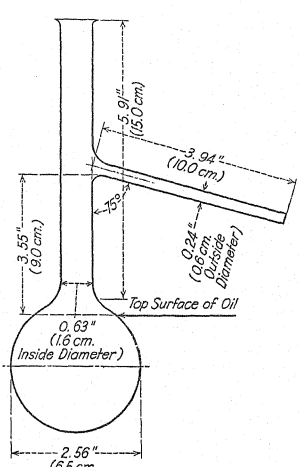


FIG. 1.—Dimensions of 100-ml. Engler Flask.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants.

[2] Prior to adoption as standard, this method was published as tentative from 1921 to 1927, being revised in 1923, 1924, and 1925.

the surface of the liquid when the flask contains its charge of 100 ml. The tube shall be approximately in the middle of the neck and set at an angle of 75 ± 3 deg. with the vertical.

(*b*) *Condenser.*—A condenser (Fig. 2 shows one of the approved types) $\frac{9}{16}$ in. (14.29 mm.) in outside diameter made of No. 20 Stubbs gage seamless brass tubing, 22 in. (55.88 cm.) in length. It shall be set so that approximately 15.5 in. (39.4 cm.) of the tube will be in contact with the cooling medium contained in the condenser jacket, subsequently described, with about 2 in. outside the cooling bath at the upper end, and 4.5 in. outside the cooling bath at the lower end. The length of tube projecting at the upper end shall be straight and shall be set at an angle of 75 deg. with the vertical. The section of the tube inside the cooling bath may be either straight or bent in any suitable continuous, smooth curve. The average gradient shall be 0.259 in. per linear inch of condenser tube (sine of angle of 15 deg.) and no section of the immersed portion of the condenser tube shall have a gradient less than 0.24 in. nor more than 0.28 in. per linear inch of tube. The projecting lower portion of the condenser tube shall be curved downward for a length of 3 in. (7.62 cm.) and slightly backward so as to insure contact with the wall of the receiving graduate at a point approximately 1 to $1\frac{1}{4}$ in. (2.54 to 3.18 cm.) below the top of the graduate when it is in position to receive the distillate. The lower end of the condenser tube shall be cut off at an acute angle. The capacity of the cooling bath shall be not less than 340 cu.in. (5.55 liters) of cooling medium. The arrangement of the tube in the cooling bath shall be such that its center line shall be not less than $1\frac{1}{4}$ in. below the plane of the top of the bath at its point of entrance and not less than $\frac{3}{4}$ in. above the floor of the bath

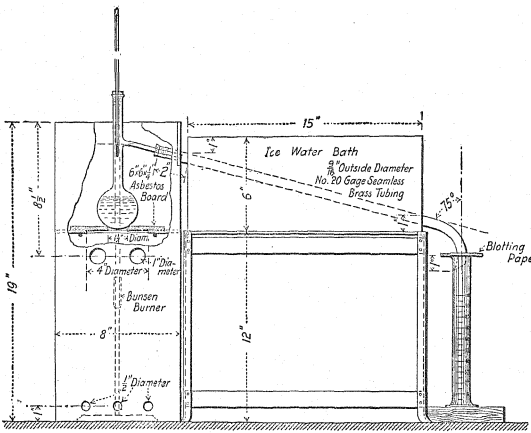


FIG. 2.—Apparatus for Distillation Test.

at its exit. Clearances between the condenser tube and the walls of the bath shall be at least $\frac{1}{2}$ in. except for the sections adjacent to the points of entrance and exit. Multiple installations are permissible, provided they conform to the dimensional requirements and the capacity of the bath is not less than 340 cu.in. per tube.

(*c*) *Shield.*—A shield (Fig. 2) 19 in. (48.26 cm.) in height, 11 in. (27.94 cm.) in length, and 8 in. (20.32 cm.) in width, made of sheet metal of approximately No. 22 gage, with a door on one narrow side having two openings 1 in. (2.54 cm.) in diameter equally spaced in each of the two narrow sides, and with a slot cut in one side for the vapor tube. The centers of these four openings shall be $8\frac{1}{2}$ in. (21.59 cm.) below the top of the shield. There shall also be three $\frac{1}{2}$-in. (1.27-cm.) holes in each of the four sides with their centers 1 in. (2.54 cm.) above the base of the shield.

(*d*) *Ring Support and Asbestos Boards.*—A ring support of the ordinary laboratory type, 4 in. (10.16 cm.) or larger in diameter, and supported on a stand inside the shield. Two hard asbestos boards, one 6 by 6 by $\frac{1}{4}$ in. (15.24 by 15.24 cm. by 6.35 mm.) with a hole $1\frac{1}{4}$ in. (3.18 cm.)[3] in diameter in its center, the sides of which shall be perpendicular to the surface; the other, an asbestos board to fit tightly inside the shield, with an opening 4 in. (10.16 cm.) in diameter concentric with the ring support. These shall be arranged as follows: The second asbestos board shall be placed on the ring and the first or smaller asbestos board on top so that it may be moved in accordance with the directions for placing the distilling flask. Direct heat shall be applied to the flask only through the $1\frac{1}{4}$-in. (3.18-cm.) opening in the first asbestos board.

(*e*) *Gas Burner.*—A burner so constructed that sufficient heat can be obtained to distill the product at the uniform rate specified in Section 4. The flame shall never be so large that it spreads over a circle of diameter greater than $3\frac{1}{2}$ in. (8.89 cm.) on the under surface of the asbestos board. A sensitive regulating valve and gas pressure governor are desirable adjuncts, as they give complete control of heating.

(*f*) *Electric Heater.*—An electric heater may be used instead of a gas burner and shall be capable of bringing over the first drop within the time specified in Section 4 (*a*) when started cold, and of continuing the distillation at the uniform rate. The electric heater shall be fitted with an asbestos board top $\frac{1}{8}$ to $\frac{1}{4}$ in. (3.18 to 6.35 mm.) in thickness, having a hole $1\frac{1}{4}$ in. (3.18 cm.) in diameter in the center. When an electric heater is employed, the portion of the shield above the asbestos board shall be the same as with the gas burner but the part below the board may be omitted.

(*g*) *Thermometer.*—An A.S.T.M. Low Distillation Thermometer, total immersion, graduated in either Centigrade or Fahrenheit degrees, having a range of 0 to 300 C. or 30 to 580 F. and conforming to the requirements for this thermometer as prescribed in the Standard Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1) of the American Society for Testing Materials.[4]

(*h*) *Thermometer.*—An A.S.T.M. High Distillation Thermometer, total immersion, graduated in either Centigrade or Fahrenheit degrees as specified, having a range of 0 to 400 C. or 30 to 760 F. and conforming to the requirements for this thermometer as prescribed in the Standard

[3] When distilling petroleum products having an end point above 470 F. (243.34 C.), the hole in the asbestos board shall be $1\frac{1}{2}$ in. (3.81 cm.) in diameter.

[4] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1) of the American Society for Testing Materials.[4]

(*i*) *Graduate.*—A graduate of the cylindrical type, of uniform diameter, with a pressed or molded base and a lipped top. The cylinder shall be graduated to contain 100 ml., and the graduated portion shall be not less than 7 in. (17.78 cm.) nor more than 8 in. (20.32 cm.) in length. It shall be graduated in single milliliters and each fifth mark shall be distinguished by a longer line. It shall be numbered from the bottom up at intervals of 10 ml. The over-all height of the graduate shall be not less than 9¾ in. (24.8 cm.), nor more than 10¼ in. (26.0 cm.). The graduations shall not be in error by more than 1 ml. at any point on the scale.

Preparation of Apparatus

3. (*a*) The condenser bath shall be filled with cracked ice,[5] and enough water added to cover the condenser tube. The temperature shall be maintained between 32 and 40 F. (0 and 4.45 C.).

(*b*) The condenser tube shall be swabbed to remove any liquid remaining from the previous test. A piece of soft, lint-free cloth attached to a cord or copper wire may be used for this purpose.

(*c*) A 100-ml. sample shall be measured in the 100-ml. graduated cylinder at 55 to 65 F. (12.8 to 18.3 C.) and transferred directly to the distillation flask. None of the liquid shall be permitted to flow into the vapor tube.

(*d*) The thermometer (Note 3) provided with a cork shall be fitted tightly into the flask so that it will be in the middle of the neck and so that the lower end of the capillary tube is on a level with the inside of the bottom of the vapor outlet tube at its junction with the neck of the flask. The thermometer shall be approximately at room temperature when placed in the flask.

NOTE 3.—For products having end points not higher than 482 F. (250 C.) *or* initial boiling points not higher than 212 F. (100 C.), the Low-Distillation Thermometer shall be used; for products having end points higher than 482 F. (250 C.) *and* initial boiling points higher than 212 F. (100 C.) the High-Distillation Thermometer shall be used.

(*e*) The charged flask shall be placed in the 1½-in. (3.18-cm.) opening in the 6 by 6-in. (15.24 by 15.24-cm.) asbestos board with the vapor outlet tube inserted into the condenser tube. A tight connection may be made by means of a cork through which the vapor tube passes. The position of the flask shall be so adjusted that the vapor tube extends into the condenser tube not less than 1 in. (2.54 cm.) nor more than 2 in. (5.08 cm.).

(*f*) The graduated cylinder used in measuring the charge shall be placed, without drying, at the outlet of the condenser tube in such a position that the condenser tube shall extend into the graduate at least 1 in. (2.54 cm.) but not below the 100-ml. mark. Unless the temperature is between 55 and 65 F. (12.8 and 18.3 C.) the receiving graduate shall be immersed up to the 100-ml. mark in a transparent bath maintained between these temperatures. The top of the graduate shall be covered closely during the distillation with a piece of blotting paper or its equivalent, cut so as to fit the condenser tube tightly.

Procedure

4. (*a*) When everything is in readiness, heat shall be applied at a uniform rate, so regulated that the first drop of condensate falls from the condenser in not less than 5 nor more than 10 min. The distillation thermometer shall be read 2 min. after heat is applied and the indication recorded as the "correction temperature." This figure is of significance only in cases when there is a

[5] Any other convenient cooling medium may be used.

question as to the accuracy[6] of the initial boiling point, as subsequently determined. When the first drop falls from the end of the condenser the reading of the distillation thermometer shall be recorded as the *initial boiling point.*

(*b*) The receiving cylinder shall then be moved so that the end of the condenser tube shall touch the side of the cylinder. The heat shall then be so regulated that the distillation proceeds at a uniform rate of not less than 4 ml. per min. nor more than 5 ml. per min. The volume of distillate collected in the cylinder shall be observed and recorded, to the nearest 0.5 ml., when the mercury of the thermometer reaches each point that is a multiple of 10 C. or the Fahrenheit equivalent of this point (30 C., 40 C., 50 C., 60 C., etc., or 86 F., 104 F., 122 F., 140 F., etc.). If preferred, the reading of the distillation thermometer may be observed and recorded when the level of the distillate reaches each 10-ml. mark on the graduate. In case a product is being tested to ascertain whether or not it conforms with a given specification, all necessary observations shall be made and recorded, whether or not they are included in the series ordinarily employed by the laboratory making the test.

(*c*) No adjustment of the heat shall be made after the liquid residue in the flask is approximately 5 ml. unless the time required to bring over the last 5 ml. of distillate and reach the end point exceeds 5 min. The *end point* is the maximum temperature observed on the distillation thermometer and is usually reached after the bottom of the flask has become dry. If the bottom of the flask is not dry the operator shall record this fact.

TABLE I.

Temperature Range		Correction[a] per 10-mm. Difference in Pressure	
deg. Cent.	deg. Fahr.	deg. Cent.	deg. Fahr.
10 to 30	50 to 86	0.35	0.63
30 to 50	86 to 122	0.38	0.68
50 to 70	122 to 158	0.40	0.72
70 to 90	158 to 194	0.42	0.76
90 to 110	194 to 230	0.45	0.81
110 to 130	230 to 266	0.47	0.85
130 to 150	266 to 302	0.50	0.89
150 to 170	302 to 338	0.52	0.94
170 to 190	338 to 374	0.54	0.98
190 to 210	374 to 410	0.57	1.02
210 to 230	410 to 446	0.59	1.06
230 to 250	446 to 482	0.62	1.11
250 to 270	482 to 518	0.64	1.15
270 to 290	518 to 554	0.66	1.19
290 to 310	554 to 590	0.69	1.24
310 to 330	590 to 626	0.71	1.28
330 to 350	626 to 662	0.74	1.32
350 to 370	662 to 698	0.76	1.37
370 to 390	698 to 734	0.78	1.41
390 to 410	734 to 770	0.81	1.45

[a] To be added in case barometric pressure is below 760 mm.; to be subtracted in case barometric pressure is above 760 mm.

(*d*) In case the time required to bring over the last 5 ml. of distillate and reach the end point exceeds 5 min. the test shall be repeated and the heat shall be adjusted when the liquid residue reaches 5 ml. This adjustment may be either an increase or a decrease but must accomplish the purpose of bringing the period required to vaporize the last 5 ml. of distillate and reach the end point within the limits of 3 and 5 min.

(*e*) The total volume of the distillate collected in the receiving graduate shall be recorded as the *recovery.*

[6] The initial boiling point is one of the observations which it is customary to report as indicative of the quality of gasoline. It is a point which is very difficult to check, especially when duplicate tests are made at different room temperatures, and it has not been found practical to devise an accurate method of correcting for this unavoidable variation in operating conditions. The practical significance of initial boiling point is not regarded as sufficient to warrant requiring that tests be conducted under regulated conditions of room temperature. The following directions are included as an expedient intended to obviate some of the difficulties that develop on account of the practice of including initial boiling point limits in specifications for the purchase and sale of gasoline:

If the "correction temperature" is below 70 F. (21 C.) or above 80 F. (27 C.), the observed initial boiling point, if it is below 150 F. (66 C.), may be considered incorrect by an amount not less than one third the difference between the "correction temperature" and 75 F. (24 C.). In case there is a dispute between the purchaser and the seller, the observed initial boiling point figures obtained by all parties shall be revised according to the following formula:

$$R = O - \frac{\text{Correction Temperature (deg. Fahr.)} - 75}{3}$$

where R = the revised initial boiling point, and
O = the observed initial boiling point.

If all of the revised figures fall within the specification limit the gasoline in question shall be considered as passing; otherwise arrangements shall, if possible, be made to conduct a distillation test with the room temperature maintained between the limits of 70 and 80 F. (21 and 27 C.), inclusive.

(*f*) The cooled residue shall be poured from the flask into a small cylinder graduated in 0.1 ml., measured when cool and the volume recorded as *residue*.

(*g*) The difference between 100 ml. and the sum of the recovery and the residue shall be calculated and recorded as *distillation loss*.

(*h*) The sum of the volume collected in the cylinder at any specified temperature and the distillation loss may be recorded as the percentage evaporated at the temperature in question. Clear distinction shall be made between the inclusion or exclusion of the distillation loss figure in the report of the volume of distillate obtained at any prescribed temperature. In the first case the figure shall be reported as the volume *evaporated*, in the second, as the volume *recovered*.

Reproducibility of Results

5. With proper care and strict attention to details, duplicate results obtained for initial boiling point and end point, respectively, should not differ from each other by more than 6 F. (3.3 C.). Duplicate readings of the volume of distillate collected in the cylinder when each of the prescribed temperature points is reached should not differ from each other by more than 2 ml. In case observations are made on the basis of prescribed percentage points, the differences in temperature readings should not exceed the amounts equivalent to 2 ml. of distillate at each point in question.

Correction for Barometric Pressure

6. The actual barometric pressure shall be ascertained and recorded, but no correction shall be made except in case of dispute. In such cases the temperature points shall be corrected to 760 mm. (29.92 in.), by the use of the Sydney Young equation, as follows:

For Centigrade readings:

$$C_c = 0.00012\ (760 - P)\ (273 + t_c)$$

For Fahrenheit readings:

$$C_f = 0.00012\ (760 - P)\ (460 + t_f)$$

where C_c and C_f = the corrections to be added to the observed temperature t_c or t_f respectively, and

P = the actual barometric pressure in millimeters of mercury.

Table I is a convenient approximation of the corrections as calculated by the above equation.

AMERICAN ASSOCIATION STATE HIGHWAY OFFICIALS STANDARD
A.A.S.H.O. NO.: T 48-42

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: Z11.6-1933
AMERICAN PETROLEUM INSTITUTE
STANDARD. A.P.I. NO.: 511-33

Standard Method of Test for

FLASH AND FIRE POINTS BY MEANS OF OPEN CUP[1]

A.S.T.M. Designation: D 92 – 33

ADOPTED, 1924; REVISED, 1933.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 92; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test is intended for determining the flash and fire points of all petroleum products except fuel oils and those having an open cup flash below 175 F. (79 C.).

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Cleveland Flash Cup.*—A Cleveland open cup, shown in Fig. 1, made of brass and conforming to the dimensional requirements prescribed in Table I. The beveled edge of the cup shall be at an angle of approximately 45 deg. There may be a fillet of approximately $\frac{1}{32}$ in. (0.397 cm.) in radius inside the bottom of the cup.

(*b*) *Heating Plate.*—A metal plate, Fig. 2, $\frac{1}{4}$ in. (0.635 cm.) in thickness, and 6 in. (15.24 cm.) in width for supporting the flash cup. The plate shall be of brass, cast iron, wrought iron, or steel. In the center of the plate there shall be a plane depression $\frac{1}{32}$ in. (0.079 cm.) in depth, and of just sufficient diameter to fit the cup. There shall be a circular opening $2\frac{5}{8}$ in. (5.50 cm.) in diameter, cut through the plate, centering with the center of the above-mentioned depression. The plate shall be covered with a sheet of hard asbestos board $\frac{1}{4}$ in. in thickness, and of the same shape as the metal plate. There shall be cut in the center of the asbestos board a circular hole just fitting the cup. Heat may be

TABLE I.—DIMENSIONAL REQUIREMENTS FOR CLEVELAND OPEN FLASH CUP.

	Inches			Centimeters		
	Min.	Normal	Max.	Min.	Normal	Max.
Inside diameter immediately below filling mark	$2\frac{15}{32}$	$2\frac{1}{2}$	$2\frac{17}{32}$	6.27	6.35	6.43
Outside diameter below flange	$2\frac{25}{32}$	$2\frac{13}{16}$	$2\frac{27}{32}$	6.75	6.83	6.91
Inside height from center of bottom to rim	$1\frac{15}{16}$	$1\frac{15}{16}$	$1\frac{17}{32}$	3.25	3.33	3.41
Thickness of bottom	$\frac{1}{16}$	$\frac{1}{8}$	$\frac{3}{16}$	0.28	0.32	0.36
Distance from rim to filling mark	$\frac{23}{64}$	$\frac{3}{8}$	$\frac{25}{64}$	0.91	0.95	0.99
Distance lower surface flange to bottom of cup	$1\frac{7}{32}$	$1\frac{1}{4}$	$1\frac{9}{32}$	3.10	3.18	3.26
Vertical distance upper surface flange to rim	$\frac{7}{64}$	$\frac{1}{4}$	$\frac{9}{64}$	0.28	0.32	0.36
Thickness of rim	$\frac{3}{64}$	$\frac{5}{32}$	$\frac{1}{64}$	0.20	0.24	0.28
Width of lower surface of flange	$\frac{1}{8}$	$\frac{19}{64}$	$\frac{5}{8}$	1.43	1.51	1.59

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants.

[2] Prior to adoption as standard, this method was published as tentative from 1921 to 1924, being revised in 1923 and 1924. Editorially revised and rearranged in 1939.

supplied from any convenient source. The use of a gas burner, electric heater, or alcohol lamp is permitted, but under no circumstances are products of combustion or free flame allowed to come up around the cup. The source of heat shall be centered under the opening in the plate and shall be of a type that will not produce local superheating. If a flame heater is used, it may be protected

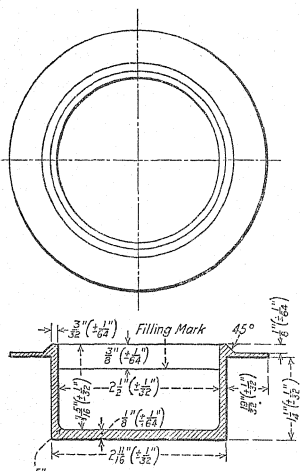


FIG. 1.—Cleveland Open Cup.

from drafts or excessive radiation by any suitable type of shield, that does not project above the level of the upper surface of the asbestos board.

(*c*) *Thermometer.*—An A.S.T.M. Open Flash Thermometer, graduated in either Centigrade or Fahrenheit degrees as specified, having a range of −6 to +400 C. or +20 to +760 F. and conforming to the requirements for this thermometer as prescribed in the Standard Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1) of the American Society for Testing Materials.[3]

Procedure

3. (*a*) The thermometer shall be suspended or held in a vertical position by any suitable device. The bottom of the bulb shall be $\frac{1}{4}$ in. (0.635 cm.)[4] from the bottom of the cup, and above a point

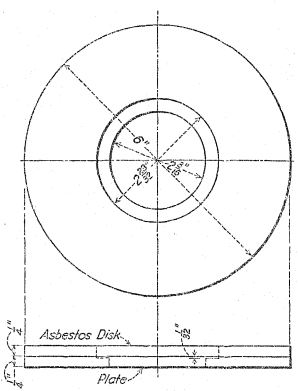


FIG. 2.—Heating Plate.

half way between the center and back of the cup.

(*b*) The cup shall be filled with the oil to be tested in such a manner that the top of the meniscus is exactly at the filling line at room temperature. When asphalt or other solid bituminous material is to be tested, it shall first be heated to a temperature not less than 300 F. (148.9 C.) nor more than 350 F., (176.7 C.) to render it sufficiently fluid.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

[4] The immersion line engraved on the thermometer stem will be $\frac{1}{16}$ in. (0.159 cm.) below the level of the rim of the cup.

The cup shall then be filled with the hot fluid at this temperature in the same manner as with oil. The subsequent procedure shall then be the same for both asphalt and solid bituminous material as with oil. The surface of the oil shall be free from bubbles. There shall be no oil above the filling line or on the outside of the apparatus.

(*c*) The test flame shall be approximately $\frac{5}{32}$ in. (0.397 cm.) in diameter.

NOTE.—For purposes of comparison it is recommended that a bead of suitable light-colored material be mounted in a convenient position so that the size of the test flame can be determined. The device for applying the flame may be of any suitable type but it is suggested that the tip be approximately $\frac{1}{16}$ in. (0.159 cm.) in diameter at the end and that the orifice be $\frac{3}{32}$ in. (0.079 cm.) in diameter. If the device for operating the test flame be mounted in such a manner as to permit automatic duplication of the sweep of the test flame the radius of swing shall be not less than 6 in.

(*d*) The test flame shall be applied as the temperature read on the thermometer reaches each successive 5 F. (2.8 C.) mark. The flame shall pass in a straight line (or on the circumference of a circle having a radius of at least 6 in.) across the center of the cup and at right angles to the diameter passing through the thermometer. The test flame shall, while passing across the surface of the oil, be in the plane of the upper edge of the cup. The time for the passage of the test flame across the cup shall be approximately 1 sec.

(*e*) The oil shall be heated at a rate not exceeding 30 F. (16.7 C.) per min. temperature rise, till a point is reached approximately 100 F. (55.6 C.) below the probable flash point of the oil. Thereafter the rate of heating shall be decreased and for at least the last 50 F. (27.8 C.) before the flash point is reached, the rate shall be not less than 9 F. (5 C.) nor more than 11 F. (6.1 C.) per min.

Flash Point

4. The flash point shall be taken as the temperature read on the thermometer when a flash appears at any point on the surface of the oil. The true flash must not be confused with a bluish halo that sometimes surrounds the test flame.

Fire Point

5. After determining the flash point, the heating shall be continued at the specified rate of 9 to 11 F. (5 to 6.1 C.) per min., and application of the test flame shall be made at the specified intervals until the oil ignites and continues to burn for a period of at least 5 sec. The method of application of the flame shall be the same as for flash point. The temperature read at the time of the flame application which causes burning for a period of 5 sec. or more shall be recorded as the fire point.

Precautions

6. The flash point and fire point tests shall be made in a room or compartment free from air drafts. The operator shall avoid breathing over the surface of the oil. It is desirable that the room or compartment be darkened sufficiently so that the flash may be readily discernible.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: Z11.24-1936

AMERICAN PETROLEUM INSTITUTE
STANDARD. A.P.I. NO.: 509-36

Standard Method of Test for

FLASH POINT BY MEANS OF THE TAG CLOSED TESTER[1]

A.S.T.M. Designation: D 56 – 36

ADOPTED, 1919; REVISED, 1921, 1936.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 56; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. (*a*) This method of test is intended for determining the flash point of all mobile liquids flashing below 175 F. (79 C.), with the exception of products classed as fuel oil which are preferably tested with the A.S.T.M. Pensky-Martens closed tester.[3]

(*b*) Provision is made in Sections 7 and 8 of this method for determining the flash point of lacquer solvents or diluents of low flash points.

Apparatus

2. (*a*) *Tag Closed Tester.*—The Tag closed tester, a diagram of which appears in Fig. 1, shall conform to the dimensional requirements prescribed in Table I.

The plane of underside of cover shall be between the top and bottom of the burner tip when the tip is fully depressed.

(*b*) *Thermometers.*—Two thermometers are required, one for measuring the temperature of the sample under test, the other for measuring the temperature of the bath. Two A.S.T.M. P.M. and Tag Thermometers, graduated in either Centigrade or Fahrenheit degrees as specified, having a range of −7 to +110 C. or +20 to +230 F. and conforming to the requirements for this thermometer as prescribed in the Standard Specifications for A.S.T.M. Thermometers

TABLE I.—DIMENSIONAL REQUIREMENTS OF TAG CLOSED TESTER.

	Inches			Centimeters		
	Min.	Normal	Max.	Min.	Normal	Max.
Depth of water surface below top of cup	1/64	1/32	1/64	2.74	2.78	2.82
Depth of oil surface below top of cup	1/64	1/32	1 1/64	2.90	2.94	2.98
Depth of top of bulb of oil thermometer when in place below top of cup	1/64	1 5/64	1 1/64	3.25	3.33	3.41
Inside diameter of oil cup at top	2 1/16	2 1/8	2 1/16	5.39	5.40	5.41
Diameter of bead on top of cover	3/64	1/16	1/64	0.36	0.40	0.44
Weight of oil cup					67 to 69 g.	

[1] Under the standardization procedure of the Society, this method is under the joint jurisdiction of A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products, and Committee D-2 on Petroleum Products and Lubricants.

[2] Prior to adoption as standard, this method was published as tentative from 1918 to 1919, being revised in 1919. Editorially revised and rearranged in 1939.

[3] See the Standard Method of Test for Flash Point by Means of the Pensky-Martens Closed Tester, (A.S.T.M. Designation: D 93) which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

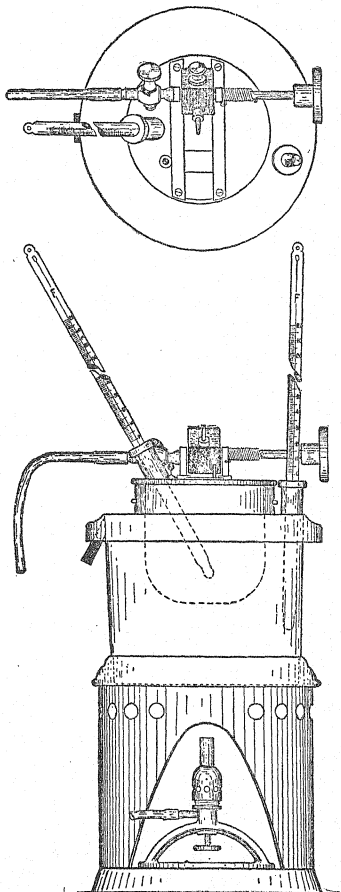


FIG. 1.—A.S.T.M. Tag Closed Tester (Arranged for Use of Gas).

(A.S.T.M. Designation: E 1) of the American Society for Testing Materials[4] shall be used.

Procedure

3. (*a*) The test shall be performed in a room or compartment darkened sufficiently to permit ready detection of the flash.

(*b*) Care shall be taken to have the tester level and steady. It shall be surrounded on three sides by an enclosure for protection from drafts. (A shield 18 in. square and 24 in. in height, open in front, is suggested. Tests made in a laboratory hood or near ventilators are not to be relied upon.)

(*c*) Gas may be used for the test flame and for heating the water bath. If gas is not available for the test flame, a wick of cotton cord may be inserted in the burner tip, a small quantity of cotton waste placed in the oil chamber to which the burner tip is attached and the chamber filled with signal, sperm, or lard oil. An alcohol lamp may be used for heating the water bath as a substitute for gas.

(*d*) The water bath thermometer shall be placed in the collar provided for it and the bath filled with water until it overflows. The temperature of the water in the bath shall be such that when testing is started it will be at least 20 F. (11 C.) below the probable flash point of the oil to be tested.

(*e*) The oil cup shall be placed in its proper position in the water bath and 50 ml. of the oil to be tested shall be measured into it, using an accurate graduate or other measuring device for the purpose. The temperature of the oil shall be at least 20 F. (11 C.) below its probable flash point when the test is started. Air bubbles on the surface of the oil shall be destroyed, and the cover with the flash point thermometer in place shall then be properly attached to the bath collar. The test flame shall be lighted, the flame being adjusted to the size of the small white bead on the cover.

(*f*) The gas burner or alcohol lamp shall be centrally placed in the base of the tester and lighted. The flame shall be so adjusted that the temperature of the oil in the cup rises at the rate of 1.8 F. (1 C.) per min. as closely as possible, but in any case not faster than 2 F. (1.1 C.) nor slower than 1.6 F. (0.9 C.) per min.

(*g*) The barometric pressure shall be recorded. If a barometer is not available, the figure may be obtained from the nearest Weather Bureau Station and an appropriate correction made for difference in altitude between such station and the laboratory.

(*h*) The initial temperature of the oil shall be recorded.

(*i*) When the temperature of the oil is 9 F. (5 C.) below its probable flash point, the knob on the cover shall be turned in such a manner as to introduce the test flame into the vapor space of the cup, and *immediately* turned back again. The time consumed in turning the knob down and back shall be about one full second, or the time required to pronounce distinctly the words "thousand and one."

(*j*) The time at which the first introduction of the test flame is made and the temperature of the oil shall be recorded.

(*k*) The application of the test flame shall be repeated after each 1 F. (0.5 C.) rise in temperature of the oil until a distinct flash in the interior of the cup is observed. The true flash must not be confused with the bluish halo which sometimes surrounds the test flame during applications immediately preceding the actual flash.

(*l*) The time and the temperature of the oil when the flash point is reached shall be recorded.

[4] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Repeat Tests

4. (*a*) If the rise in temperature of the oil from the time of making the first introduction of the test flame to the time at which the flash point is observed was more rapid than 2 F. (1.1 C.) per min., or slower than 1.6 F. (0.9 C.) per min., the test shall be repeated, adjusting the gas burner or alcohol lamp to the proper rate of heating.

(*b*) It is not necessary to turn off the test flame with the small regulating valve on the cover; it may be left adjusted to the proper size of flame.

(*c*) After completing the preliminary test to determine the approximate flash point, the burner or lamp shall be removed, the oil cover lifted and the thermometer bulb carefully wiped off. The oil cup shall be removed, emptied, and carefully wiped until dry.

(*d*) The temperature of the bath shall be lowered by the addition of cold water until it is 15 F. (8 C.) below the flash point of the oil as shown by the preliminary test.

(*e*) The oil cup shall be replaced and a fresh 50-ml. sample measured into it. The test procedure, as described in Section 3, shall then be repeated, introducing the test flame for the first time, however, when the oil temperature is 10 F. (5.5 C.) below the flash point obtained in the preliminary test.

(*f*) Oil which has once been subjected to the flash test shall be discarded.

(*g*) If test is to be repeated, a fresh sample shall be used.

Average Value of Flash Point

5. If two or more determinations agree within 1 F. (0.5 C.), the average of these results, corrected for barometric pressure, shall be considered the flash point. If two determinations do not check within 1 F. (0.5 C.), a third determination shall be made and, if the maximum variation of the three tests is not greater than 2 F. (1 C.), their average, after correcting for barometric pressure, shall be considered the flash point.

Correction for Barometric Pressure

6. Correction for barometric pressure shall be made only in cases of dispute or when the barometer reading varies more than $\frac{1}{2}$ in. (13 mm.) from the standard pressure of 29.92 in. (760 mm.). When the barometer reading is below this standard pressure, add to the thermometer reading 1.6 F. (0.9 C.) for each 1 in. (25 mm.) of barometer difference to obtain the true flash point. When the barometer reading is above the standard pressure, deduct 1.6 F. (0.9 C.) for each 1 in. (25 mm.) of barometer difference to obtain the true flash point.

Determination of Flash Point of Lacquer Solvents or Diluents of Low Flash Point

Apparatus

7. The apparatus shall consist of the following:

(*a*) *Flash Point Tester.*—As described in Section 2 (*a*).

(*b*) *Thermometer.*—For determining flash points below 40 F. (4.4 C.), suitable thermometers of low range shall be used. These thermometers shall conform to the requirements specified in Section 2 (*b*) except that the ranges shall be 0 F. to 120 F. or −20 C. to +50 C. These thermometers may not be used in the testing of materials flashing above 70 F. (21.1 C.).

Procedure

8. The procedure to be followed shall be the same as that described in Sections 3 to 6, except that in determining flash points below 40 F. (4.4 C.) the bath shall be filled with brine or other low-freezing liquid instead of water.

Standard Specifications for
DRY BLEACHED SHELLAC[1]

A.S.T.M. Designation: D 207 – 35

ADOPTED, 1933; REVISED, 1935.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 207; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover two grades of dry bleached shellac, namely, regular and refined.

NOTE.—Dry bleached shellac is white shellac. It is known as "bone dry," "vacdry," or "kiln-dried" bleached shellac. The refined grade is bleached shellac that has been treated in the process of manufacture so as to remove practically all wax and matter insoluble in hot alcohol.

Properties

2. Dry bleached shellac shall conform to the following requirements:

	Regular	Refined
Iodine number, max.	10	10
Matter insoluble in specified hot solvents, max., per cent.	1.0	0.2
Moisture, max., per cent.	5.0	5.0
Wax, max., per cent	5.5	0.2
Matter soluble in water, max., per cent.	1.0	0.3
Ash, max., per cent.	1.0	0.5

Methods of Testing

3. The material shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the Standard Methods of Sampling and Analysis of Shellac (A.S.T.M. Designation: D 29) of the American Society for Testing Materials.[3]

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to their present adoption as standard, these specifications were published as tentative from 1925 to 1926. They were adopted in 1926, but withdrawn and replaced in 1930 by D 207 – 29 T which were published as tentative from 1929 to 1933, being revised in 1933.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for
SHELLAC VARNISHES[1]

A.S.T.M. Designation: D 360 – 41

ADOPTED, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 360; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. Shellac varnish is a solution or "cut" of a specified grade of pure orange shellac or of dry bleached shellac (the manufactured products from the secretion of the *Laccifer Lacca Kerr*, with no other added resin, copal, or other adulterants) in the most commonly used solvent, 95 per cent (190-proof) specially denatured alcohol, Formula No. 1 of the U. S. Internal Revenue Bureau, or in other specified denatured alcohol or solvent.

Types

2. Shellac varnish shall be furnished as Orange, grades A and B, and as Bleached, grades "Regular" and "Refined." Each type shall be furnished as very light, light, medium, heavy, very heavy, or extra heavy body, as specified, or of any other body mutually agreed upon by the purchaser and the seller.

Color

3. The color of shellac varnish shall be equal to the color of a standard varnish of similar concentration mutually agreed upon by the purchaser and the seller.

NOTE.—Attention is called to the fact that the purchaser and the seller must agree upon one of the two methods for determining color appearing in the Standard Methods of Sampling and Analysis of Shellac (A.S.T.M. Designation: D 29) of the American Society for Testing Materials.[3]

Copal

4. All pure shellac varnishes shall not contain gum copal.

Drying Time

5. Shellac varnish shall dry hard in 1 hr. Shellac varnishes having a body greater than that for a light body varnish shall be thinned to the concentration of the light varnish. Special

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1933 to 1934, being revised in 1934. They were adopted in 1934, published as standard from 1934 to 1939, but withdrawn, revised, and republished as tentative in 1939.

These specifications comprise a consolidation of, and replace the former Standard Specifications for Shellac Varnish (D 359 – 35), and for Centrifuged Shellac Varnish (D 360 – 34), which specifications were accordingly discontinued in 1939.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

conditions for this test may be made by mutual agreement between the purchaser and the seller.

Body

6. The body of the respective types shall be based upon the percentage of nonvolatile matter, and shall conform to the following minimum requirements:

	Nonvolatile Matter, min., per cent	
	Orange, Grades A and B	Bleached, Regular and Refined
Very light body (3-lb. cut) varnish	28.5	28.0
Light body (4-lb. cut) varnish	35.0	34.5
Medium body (4.5-lb. cut) varnish	37.5	37.0
Heavy body (5-lb. cut) varnish	40.0	39.5
Very heavy body (6-lb. cut) varnish	44.0	43.5
Extra heavy body (8-lb. cut) varnish	51.0	50.5

NOTE.—The term "cut" is defined as the number of pounds of dry shellac (containing only that amount of moisture acceptable for proper cutting with the solvent to insure a satisfactory varnish) that were added to 1 gal. of specified solvent in manufacturing the varnish.

Allowance has, therefore, been made for 2 per cent maximum moisture and other volatile matter and 3 per cent insoluble matter in orange shellac, and for 6 per cent moisture and other volatile matter in bleached shellac, in arriving at a minimum value. Varnishes of the above specified bodies or "cuts" should accordingly conform to the respective minimum requirements specified for nonvolatile matter.

Properties

7. The nonvolatile matter in the varnish shall conform to the following requirements:

	Orange		Bleached	
	Grade A	Grade B	Regular	Refined (Note)
Iodine number, max	...	24.5	10.0	10.0
Matter insoluble in specified hot solvents, max., per cent	1.0	1.0	1.0	0.2
Wax, max., per cent	5.5	5.5	5.5	0.2
Ash, max., per cent	0.3	0.3	1.0	0.5

NOTE.—Refined bleached shellac varnish is not centrifuged but, in commercial practice, is clarified by settling or filter pressing with or without the addition of oxalic acid or other coagulating precipitating medium; therefore, this product, known in the trade as White French Varnish, is practically free of wax, alcohol insoluble matter, and ash.

Packing and Marking

8. Shellac varnish shall be delivered in standard commercial containers. Each container shall be marked "pure."

Methods of Testing

9. The sampling and methods of testing shall be conducted in accordance with the Standard Methods of Sampling and Analysis of Shellac (A.S.T.M. Designation: D 29) of the American Society for Testing Materials.[3]

Standard Specifications for

LIQUID PAINT DRIERS[1]

A.S.T.M. Designation: D 600 – 43

ADOPTED, 1943.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 600; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover metallic driers in liquid form for use in paints and similar finishing materials. Two classes are covered, as follows:

Class A.—Driers of the resinate or linoleate types, dispersed in oils, resins, and petroleum spirits or turpentine.

Class B.—Driers of the newer soluble type, which are metallic salts of several types of organic acids, dispersed in suitable media, and usually free of resins.

Composition and Properties

2. (*a*) *General Requirements.*—The drier shall conform to the general requirements as to composition and properties for the class specified in Table I.

TABLE I.—REQUIREMENTS FOR LIQUID PAINT DRIERS.

Solid or suspended matter, max., per cent	Class A	1.0
	Class B	0.1
Color[a]	not darker than a freshly prepared solution[b] of 1.28 g. of $K_2Cr_2O_7$ in 100 ml. of H_2SO_4 (sp. gr. 1.84)	
Miscibility with oil[c]	miscible with 9 volumes of pure raw linseed oil[d], without visible sediment or curdling at the time of mixing	

[a] This requirement applies to all driers except those containing iron.

[b] This solution is equivalent to the following color standards:

		Color Number
Gardner color standards (1933)		16
Hellige color comparator		8L
Lovibond equivalent	red	26.2
	yellow	140.0
	blue	0.3

[c] In the case of disagreement between the seller and the purchaser, the test for miscibility with oil shall be made at 25 ± 1 C.

[d] The linseed oil shall conform to the Standard Specifications for Raw Linseed Oil (A.S.T.M. Designation: D 234).[3]

(*b*) *Metallic Content.*—The drier shall conform to the requirements as to metallic content for the type and class specified in Table II.

NOTE.—The metallic contents specified in Table II represent the normal amounts required for a drier that will be used with linseed oil in the ratio of 1 volume of drier to 19 volumes of oil.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1941 to 1943, being revised in 1943.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

TABLE II.—REQUIREMENTS FOR METALLIC CONTENT OF LIQUID PAINT DRIERS.

Type	Metallic Content of Drier	Class A	Class B
Lead and Manganese	Lead, min., per cent	2.5	2.5
	Manganese, min., per cent	0.6	0.6
Lead	Lead, min., per cent	5.0	5.0
Lead and Cobalt	Lead, min., per cent	2.5	2.5
	Cobalt, min., per cent	0.25	0.25
Manganese	Manganese, min., per cent	1.0	1.0
Cobalt	Cobalt, min., per cent	0.6	0.6
Zinc	Zinc, min., per cent	...	[a]
Zinc and Cobalt	Zinc, min., per cent	...	[a]
	Cobalt, min., per cent	...	[a]
Iron	Iron, min., per cent	[a]	[a]

[a] As agreed upon by the seller and the purchaser.

Methods of Testing

3. The properties enumerated in these specifications shall be determined in accordance with the Standard Methods of Testing Liquid Driers (A.S.T.M. Designation: D 564),[3] except that in the test for miscibility, the sample shall be mixed with 9 volumes of linseed oil instead of 19 volumes.

Standard Methods of

SAMPLING AND ANALYSIS OF SHELLAC[1]

A.S.T.M. Designation: D 29 - 40

ADOPTED, 1917; REVISED, 1924, 1925, 1928, 1933, 1939, 1940.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 29; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods cover the procedures for sampling and analyzing dry shellac and shellac varnish.

DRY SHELLAC

SAMPLING

Orange Shellac

2. (*a*) Only original packages of shellac shall be sampled. Samples, preferably from not less than 10 per cent of the lot, shall be drawn either by hand or by means of a suitable tryer.

(*b*) *Free Shellac.*—In sampling "free" shellac, approximately double handfuls shall be taken from each bag of shellac selected for sampling. The total amount of shellac taken shall then be reduced by quartering to 2 lb. After quartering, the 2-lb. sample of shellac shall be ground so that 100 per cent will pass a No. 20 (840-micron) sieve. This 20-mesh ground shellac shall again be quartered to 0.5 lb., and this quantity shall be ground so that 100 per cent will pass a No. 40 (420-micron) sieve and then thoroughly rolled. One half of this sample shall be reserved by the purchaser and the other half used for the analysis.

(*c*) *Blocky or Matted Shellac.*—In sampling "blocky" or "matted" (hard) shellac, approximately 1 lb. shall be taken from each bag selected for sampling. The total amount of shellac taken shall be ground to pass a $\frac{1}{4}$-in. sieve and then reduced by quartering to 2 lb. After quartering, the 2-lb. sample of shellac shall be ground so that 100 per cent will pass a No. 20 (840-micron) sieve. This 20-mesh ground shellac shall again be quartered to 0.5 lb., and this quantity shall be ground so that 100 per cent will pass a No. 40 (420-micron) sieve and then thoroughly rolled. One half of this sample shall be reserved by the purchaser and the other half used for analysis.

Bleached Shellac

3. *Bone-dry Shellac.*—(*a*) Only original, unopened barrels shall be sampled.

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these methods were published as tentative from 1914 to 1917, being revised in 1915 and 1916.

A "tentative revision" of this standard appears in the Tentative Revisions of Standards section at the back of this book.

(*b*) Samples shall be taken either with a scoop or grain sampler from different parts of each barrel directly after the barrels are opened or bored, from 20 per cent of any lot, but from not less than two barrels in a lot.

(*c*) These samples taken from each barrel shall amount to approximately 1 lb., and the composite sample from all the barrels shall then be well mixed and where larger than 3 lb. shall be reduced by quartering to a sample of this size. This sample shall then be divided into two equal portions and placed in two clean, dry quart jars or cans with tight caps or tops. The jars or cans shall be filled and the tops then sealed with paraffin wax or beeswax.

(*d*) The sample of shellac when received by the analyst shall then be ground to entirely pass a No. 20 (840-micron) sieve, placed in a tightly stoppered container, and thoroughly mixed by shaking the closed container so as to avoid possible loss of moisture. The necessary determinations on the sample shall be made with as little delay as possible.

(*e*) If the shellac in the barrels is blocked or run together, samples aggregating at least 1 lb. shall be chopped or chiseled from different parts of each barrel and the composite sample from all barrels shall be quickly crushed to lumps about 1 in. square and smaller. This shall then be well mixed and where the amount is larger than 3 lb. reduced by quartering to a sample of approximately this size. This sample shall then be divided and sealed as described in Paragraph (*c*).

(*f*) The following information shall be legibly marked on a label attached to each sample: The date of sampling, the number of barrels sampled, the total number of barrels in the lot, the condition of the barrels and contents, the manufacturer's name, and the lot or code numbers of the barrels.

(*g*) One of the samples shall be forwarded immediately to an official analyst, or to the seller; the other shall be retained intact in a cool, dry place for further disposition.

(*h*) *Hanks or Bars.*—Two or more hanks or bars shall be selected from each barrel to be sampled and the samples shall be prepared from the hanks or bars so selected the same as for bone-dry shellac as described in Paragraph (*e*).

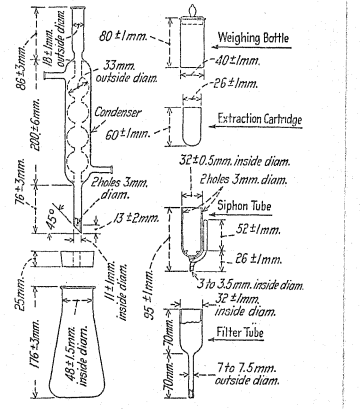


FIG. 1.—Extraction Apparatus for Insoluble Matter Test, Method A.

(*i*) *Crushed or Ground.*—Crushed or ground shellac shall be sampled the same as for bone-dry shellac as described in Paragraphs (*a*) to (*d*).

INSOLUBLE MATTER

A. For Orange, Button, Seed, and Regular Bleached Shellac.

Solvent Required

4. 95 *per cent Alcohol.*—Specially denatured, 95 per cent alcohol; Formula No. 1 or No. 30 of the U. S. Internal Revenue Bureau.

Apparatus

5. The apparatus (Figs. 1 and 2) shall consist of the following:

(*a*) *Condenser.*—A 200 ± 6 mm., four-bulb, glass condenser (Allihn type) with stem 76 ± 3 mm. in length and 11 ± 1 mm. in inside diameter. The stem shall have two holes, 3 mm. in diameter and 13 ± 2 mm. from the tip, for a wire. The tip shall be cut at an angle of 45 deg.

(*b*) *Siphon Tube.*—A Knoefler-type glass siphon tube 32 ± 0.5 mm. in inside diameter, and 52 ± 1 mm. from top of the side tube bend to the top of the three indentations on the bottom for supporting the extraction cartridge. The over-all height shall be 95 ± 1 mm.

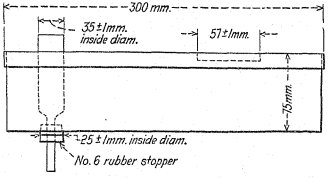


FIG. 2.—Hot-Water Bath for Insoluble Matter Test, Method A.

The siphon tube shall have two holes near the top for a wire to be fastened to a condenser tip, leaving about 6 mm. space between the top of the tube and the tip. The side discharge tube shall be 3 to 3.5 mm. in inside diameter.

(*c*) *Flask.*—A heat-resistant glass,[3] wide-mouth Erlenmeyer flask 176 ± 3 mm. in height and 48 ± 1.5 mm. in inside diameter at the top. The flask shall have a tight-fitting cork 25 mm. in depth and bored to fit the stem of the condenser. The bottom of the cork shall be just above the holes for the wire. To support the flask a suitable ring support with iron clamp and nichrome or iron gauze is required. The gauze shall contain no asbestos covering.

(*d*) *Filter Tube.*—A carbon filter tube 32 ± 1 mm. in inside diameter, with a light spiral spring on the bottom to hold up the extraction cartridge. The stem of the filter tube shall be fitted with a No. 6 rubber stopper and firmly held in a hot-water bath.

(*e*) *Extraction Cartridges.*—Extraction cartridges 26 ± 1 mm. in diameter and 60 ± 1 mm. in height (Schleicher & Schull No. 603 or equivalent).

(*f*) *Weighing Bottle.*—A glass-stoppered weighing bottle 80 ± 1 mm. in height and 40 ± 1 mm. in diameter.

(*g*) *Hot-Water Bath.*—A hot-water bath made of 26-oz. copper, 30 cm. in length, 10 cm. in width, and 7.5 cm. in depth, with cover 1.3 cm. deep (Fig. 2). The cover shall have a flanged hole 57 ± 1 mm. in diameter for a 200-ml. beaker, also a hole 35 ± 1 mm. in diameter through which the filter tube top projects. Directly below this hole, in the bottom of the bath, shall be a flanged hole 25 ± 1 mm. in diameter to hold the No. 6 rubber stopper through which the stem of the filter tube extends to discharge into the bottle or flask. The hot-water bath shall be mounted on a low iron tripod or stand.

[3] Pyrex glass is very satisfactory for this purpose.

(*h*) *Gas Burner.*—A low-form adjustable bunsen burner, carrying a draft shield by means of a star chimney support.

Preparation of Extraction Cartridge

6. Place 125 ml. of 95 per cent alcohol in the flask and a new extraction cartridge in the siphon tube. Connect to the condenser, making sure there is an ample flow of cold water through the condenser. Adjust the flame of the burner so as to give a cycle of filling and emptying in the siphon tube every 2 min., and extract for 30 min. Dry the cartridge in an air bath at 105 C. At the end of 2 hr., weigh in a weighing bottle, lifting the stopper momentarily before weighing. Continue drying, weighing as before after each hour of drying, until the loss in weight between successive weighings does not exceed 2 mg. A number of cartridges may be extracted and kept in weighing bottles or a desiccator until required. New cartridges only shall be used.

Procedure

7. Before analysis, thoroughly mix and roll the shellac in order to insure uniformity of the sample (Note 1). Weigh to the nearest 1 mg. a 5-g. ± 1-mg. sample taken directly from the mixing sheet, and place in a 200-ml. tall-form, lipped beaker; add 125 ml. of 95 per cent alcohol, and place in the hot-water bath (Fig. 2). Boil the solution vigorously for 30 min. to insure complete solution of the shellac and wax; keep the volume of alcohol constant. Meanwhile place an extracted, weighed cartridge in the filter tube. The hot water around the tube must be maintained at not less than 90 C. Wet the cartridge with hot alcohol, and decant the boiling alcohol-shellac solution into the warm cartridge until the beaker is nearly empty. Wash the remaining solution and insoluble matter into the cartridge, using a "policeman" if necessary, with successive portions of very hot 95 per cent alcohol. Finally, wash the cartridge from the top down. A complete washing will require at least 75 ml. of hot alcohol. Transfer the cartridge with insoluble matter to the extraction apparatus, and extract with 125 ml. of boiling 95 per cent alcohol for exactly 1 hr. (Note 2). Adjust the bunsen burner so that a complete filling and emptying of the siphon tube with hot alcohol requires 2 min. or 30 cycles per hour (Note 3). The condenser shall always have an ample flow of cold water so that the alcohol can be promptly condensed and returned to the flask. (The apparatus shall be guarded from draft.) Remove the cartridge, drain in an upright position, and dry in an air bath at 105 C. At the end of 2 hr. weigh in the weighing bottle, lifting the stopper momentarily to break the vacuum before weighing. Continue drying, weighing as before after each hour of drying, until the loss in weight between successive weighings does not exceed 2 mg. From the weight of the residue and the weight of the sample calculate the percentage of insoluble matter.

NOTE 1.—When the determination of matter insoluble in hot alcohol is required for hanks, bars, or ground bleached shellac, the sample shall be dried to less than 6 per cent moisture by drying overnight at room temperature. The sample shall be exposed in a thin layer and loosely covered to prevent the addition of dust.

NOTE 2.—During the 1-hr. extraction, all the remaining soluble matter should be extracted by the boiling alcohol, leaving in the cartridge all the insoluble matter.

NOTE 3.—Occasionally, lacs, including bleached shellacs, are encountered which will not yield the required 30 siphons per hour due to slow filtration. In these instances the extraction shall be continued until 30 siphons have occurred and shall be reported as "abnormal", or slow filtering.

B. For all Grades of Shellac, Including Refined Bleached Shellac.[4]

Solvent Required

8. *Normal Butyl Alcohol.*—A 98 to 99 per cent normal butyl alcohol or 98 to 99 per cent ethylene glycol monoethyl ether (beta ethoxy ethyl alcohol) conforming to the requirements of the Standard Specifications for Butanol (Normal Butyl Alcohol) (A.S.T.M. Designation: D 304)[5] or the Standard Specifications for Ethylene Glycol Monoethyl Ether (A.S.T.M. Designation: D 331) of the American Society for Testing Materials.[5]

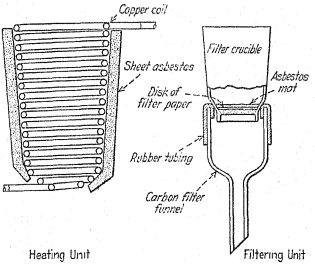


FIG. 3.—Apparatus for Insoluble Matter Test, Method B.

Apparatus

9. The apparatus (Fig. 3) shall consist of the following:

(*a*) *Crucible.*—A Jena glass-filter crucible having a capacity of 30 ml. with a fritted-glass filter disk having a thickness of 3 mm. and a porosity of 5 to 7 (average diameter of pores 35 μ to 40 μ).

(*b*) *Filter Tube.*—A carbon filter tube made of heat-resistant glass[3] to fit the crucible.

(*c*) *Heating Coil.*—A heating coil made of 3-mm. diameter copper tubing of such size and shape that the filter crucible and the large part of the carbon tube will fit snugly within it. The outside shall be insulated with sheet asbestos.

Preparation of Filtering Unit

10. Cut a disk of filter paper (Whatman No. 41 or equivalent) to fit inside the crucible and place it on top of the glass filter. Introduce upon the filter paper, in the customary manner, an asbestos mat approximately 3 mm. in thickness. Dry the crucible to constant weight at 105 to 110 C. and cool in a desiccator. Weigh the prepared crucible and place it within the carbon tube, using thin rubber tubing to form an airtight connection. Place the combined filtering unit within the heating unit, attach to a suction flask, and pass a current of steam through the coil.

Procedure

11. Weigh to the nearest 1 mg. a 5-g. ± 100-mg. sample of the dry shellac (Note 1) into a 200-ml. beaker. Add 75 ml. of the solvent and bring the solution to boiling on an electric stove. Keep the solution boiling slowly for 5 min. to insure complete solution. Pour about 10 ml. of the boiling solvent from a wash bottle into the heated crucible. Apply gentle suction and immediately pour the boiling shellac solution into the crucible in such a manner as to retain as much as possible of the insoluble residue in the beaker. Wash the insoluble residue successively with three 20-ml. portions of the solvent, boiling the solution on the electric stove for about 1 min. before each filtration. Finally, transfer the residue from the beaker to the crucible with a stream of the boiling solvent from a wash bottle, using a "policeman" when necessary. Wash down the inner walls

[4] C. C. Hartman, "Determination of Insoluble Matter in Shellac," U. S. Bureau of Standards *Journal of Research*, Vol. 7, No. 6, p. 1105 (1931).

[5] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

of the crucible with the boiling solvent. The total volume of the solvent used shall be approximately 175 ml. It is advisable to keep the crucible covered with a small watch glass at all times, except when actually transferring the solution from the beaker to the crucible, or when washing down the inside walls of the crucible. By this procedure a higher temperature is maintained within the crucible. Allow the crucible to remain inside the heating coil with the suction on for a few minutes, so as to suck it as dry as possible. Remove the crucible, wash the outside with boiling solvent, and dry it in an oven at 105 to 110 C. for 1 hr., cool in a desiccator, and weigh. From the weight of the residue and the weight of the sample calculate the percentage of insoluble matter.

NOTE 1.—For refined bleached shellac and other shellacs having a very low percentage of insoluble matter, a 10 to 20-g. sample shall be taken.

NOTE 2.—The insoluble matter can be easily removed together with the asbestos mat and filter paper. The crucible may be used several times without further cleaning. When additional cleaning is necessary it may be easily accomplished by immersing the crucible in a hot mixture of sulfuric and nitric acids for a few minutes.

IODINE NUMBER

Special Solutions Required

12. (*a*) *Acetic Acid.*—Glacial acetic acid (99 per cent) having a melting point of 14.8 C. and free from reducing impurities. If these requirements are not complied with the results of the iodine number determination will be erratic. Determine the melting point and test the acetic acid for reducing impurities as follows:

Melting Point Determination.—Fill a 6-in. test tube about two-thirds full of the acetic acid and insert into the acid an accurate thermometer (Note) by means of a cork stopper fitting the test tube. The amount of acid should be at least double the quantity required to cover the bulb of the thermometer when the bottom of the latter is ½ in. from the bottom of a test tube. Suspend this tube within a larger test tube by means of a cork. Cool the acid by immersing the assembly in ice water until the temperature is 10 C., then withdraw the assembly from the ice water and stir the acid rather vigorously for a few moments, thus causing the supercooled liquid to crystallize partially and give a mixture of liquid and solid acid. Take thermometer readings every 15 sec. and consider as the true melting point that temperature at which the reading remains constant for at least 2 min.

NOTE.—An engraved stem thermometer calibrated between 10 and 65 C. in 0.1 C. intervals and with the 0 C. point marked on the stem is recommended. The thermometer shall have an auxiliary reservoir at the upper end, a length of about 37 cm., and a diameter of about 6 mm. It is recommended that the thermometer be certified by the National Bureau of Standards. A suitable thermometer is that used for the titer test.[6]

Test for Reducing Impurities (Potassium Permanganate Test).[7]—Dilute 2 ml. of the acetic acid with 10 ml. of distilled water and 0.1 ml. of 0.1 *N* $KMnO_4$ solution and maintain at 24 ± 3 C. At the end of 2 hr. the pink color shall not be discharged.

(*b*) *Wijs Iodine Monochloride Solution.*—Dissolve 13 g. of iodine in a liter of the acetic acid, using gentle heat if necessary, determining the strength by titration with thiosulfate. Set aside 50 to 100 ml. of the solution and introduce dry chlorine gas into the remainder until the characteristic color change occurs, and the halogen content has been nearly doubled as ascertained by titration. If

[6] Standard Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

[7] *Industrial and Engineering Chemistry*, No. 18, p. 637, June, 1926.

the halogen content has been more than doubled, reduce it by adding the requisite quantity of the iodine - acetic acid solution. A slight excess of iodine does no harm but an excess of chlorine shall be avoided.

NOTE 1.—*Example.*—If the titration of 20 ml. of original iodine - acetic acid solution required 22 ml. of 0.1 *N* thiosulfate, 20 ml. of the finished Wijs solution should require between 43 and 44 ml. of the thiosulfate solution.

NOTE 2.—The Wijs solution should be tested against an orange shellac the iodine number of which is accurately known.[8] The iodine number thus obtained should be within plus or minus 0.5 of the known iodine number.

(*c*) *Chloroform.*—U. S. P. grade, acetone-free.

(*d*) *Standard Sodium Thiosulfate Solution.*—Dissolve pure sodium thiosulfate in distilled water that has been well boiled to free it from carbon dioxide, in the proportion so that 24.83 g. of $Na_2S_2O_3 \cdot 5H_2O$ will be present in 1000 ml. of the solution. It is best to let this solution stand for about two weeks before standardizing. Standardize with pure resublimed iodine or potassium biiodate.[9] This solution will be approximately 0.1 *N* and it is best to leave it as it is after determining its exact iodine value, rather than to attempt to adjust it to exactly 0.1 *N* strength. Preserve in a stock bottle with a guard tube filled with soda lime.

(*e*) *Starch Solution.*[10]—Make a paste of 0.2 g. of soluble (potato) starch in cold water and pour into 100 ml. of boiling water, boil for 5 min., cool, and bottle.

(*f*) *Potassium Iodide Solution.*—Prepare fresh by dissolving 10 g. of KI, free from iodate, in 90 ml. of distilled water.

Procedure

13. (*a*) Introduce a 0.2-g. sample (Notes 1 and 2) of finely-ground shellac into a 250-ml., dry, clear glass bottle having a ground-glass stopper. Add 20 ml. of glacial acetic acid and twirl the bottle gently on top of a hot-water bath at 65 to 70 C. until solution is complete, except for the wax. This should not require more than 15 min. A pure shellac is rather difficultly soluble; solution is quicker according to the percentage of rosin present. Add 10 ml. of acetone-free chloroform and cool the solution to from 21.5 to 22.5 C. Allow the bottle to stand for at least 30 min. half immersed in a shallow pan of water at a temperature of 21.5 to 22.5 C., well insulated or equipped with a suitable thermostat, before adding the Wijs solution. Add 20 ml. of the Wijs solution (Note 3), which shall be at a temperature of 21.5 to 22.5 C., from a pipette having a rather small delivery aperture (about 30 sec.). Close the bottle, place it again into the pan of water, and note the time. The bottle shall be kept half immersed in water at from 21.5 to 22.5 C. during the hour that the shellac is exposed to the Wijs solution. Twirl the bottle occasionally during the hour. After exactly 1 hr., add 10 ml. of freshly prepared KI solution, washing into the bottle any Wijs solution on the stopper with the same. Titrate the solution immediately with about 25 to 30 ml. of the 0.1 *N* $Na_2S_2O_3$, allow the solution to run in rapidly, and shake vigorously until the solution becomes a straw color. Now add 15 ml. of freshly prepared starch solution and slowly finish titrating. The end point is sharp; disregard any color returning after about 30 sec.

(*b*) *Blank.*—Run a blank determination on the reagents at the same time. The blank is necessary because of the well known effect of temperature changes on the volume, and possible loss of strength of the Wijs solution.

(*c*) *Standard Shellac.*[8]—With every set

[8] Standard samples of pure orange shellac, rosin-free, of accurately known iodine number may be purchased from the Secretary of the U. S. Shellac Importers' Assn., 155 John St., New York City.

[9] "Analytical Chemistry," Treadwell and Hall, Vol. II, Sixth Edition, pp. 551 and 553

[10] Editorially revised in 1941.

of tests, run a determination on a sample of pure shellac of known iodine value.

NOTE 1.—In the case of grossly adulterated samples, or in the testing of pure rosin, it is necessary to use, instead of 0.2 g. of material, a smaller quantity (0.15 or 0.1 g.) in order that the excess of iodine monochloride may not be too greatly reduced since the excess of halogen is one of the factors in determining the amount of absorption. In case less than 25 ml. of the $Na_2S_2O_3$ is required, another test shall be made, using a smaller quantity of the shellac.

NOTE 2.—In weighing shellac, some difficulty is at times experienced on account of its electrical properties. In very dry weather it may be found that the necessary handling to prepare it for weighing has electrified it, and that it may be necessary to leave it in the balance pan at rest for a few minutes before determining the weight.

NOTE 3.—If a number of samples are being run, at least 5 min. shall be allowed between the additions of the Wijs solution.

Calculation

14. The iodine number of the sample of shellac tested shall be calculated from the difference between the blank titration and the titration of the sample, and the iodine value of the $Na_2S_2O_3$ solution. (Iodine number is given in centigrams of iodine to 1 g. of sample.)

PURITY

Rosin

15. *Qualitative Test for Rosin.*—Add 20 ml. of absolute alcohol or glacial acetic acid (melting point 13 to 15 C.) to 2 g. of the shellac and thoroughly dissolve. Add 100 ml. of petroleum ether and mix thoroughly. Add approximately 2 liters of water and separate a portion of the ether layer (at least 50 ml.) and filter if cloudy. Evaporate the petroleum ether and test the residue with the Halphen-Hicks reagent[11] as follows:

Solution A.—One part by volume of phenol dissolved in two parts by volume of carbon tetrachloride.

Solution B.—One part by volume of bromine dissolved in four parts by volume of carbon tetrachloride.

Add 1 to 2 ml. of Solution *A* to residue left after evaporation of the petroleum ether solution and pour this mixture into a cavity of an ordinary porcelain color-reaction plate until it just fills the depression. Immediately fill an adjacent cavity with Solution *B*. Cover the plate with an inverted watch glass and note the color, if any, produced in the former solution by the action of the bromine vapors from Solution *B*. A decided purple or deep indigo blue color is an indication of the presence of rosin.

Copal

16. *Qualitative Test for Copal.*—Make an approximately 35 per cent solution of the shellac in 95 per cent denatured alcohol, then filter. To 10 ml. of this filtrate in a large test tube (6 by $\frac{3}{4}$ in.), add 99 per cent methyl alcohol to nearly fill the tube, and mix thoroughly. The formation of a precipitate after standing is an indication of copal. Shellac free from copal should remain clear.

Estimation of Adulterant

17. Since the variation between the highest and lowest iodine numbers of a pure shellac is not great, it is recommended that the following assumptions (Note) be made:

	Assumed Iodine Number
Rosin-free and copal-free orange shellac	18
Rosin-free and copal-free bleached shellac	10
Rosin	228
Copal	130

The percentages of adulterants shall be calculated as follows:

$$\text{Percentage of rosin in orange shellac} = \frac{x - 18}{228 - 18} \times 100$$

[11] *Industrial and Engineering Chemistry*, Vol. 3, p. (1911).

$$\text{Percentage of rosin in bleached shellac} = \frac{x - 10}{228 - 10} \times 100$$

$$\text{Percentage of copal in orange shellac} = \frac{x - 18}{130 - 18} \times 100$$

$$\text{Percentage of copal in bleached shellac} = \frac{x - 10}{130 - 10} \times 100$$

where:

x = iodine number of the sample under test, determined in accordance with Sections 13 and 14.

NOTE.—The results obtained by assuming the values of 18 and 10 as the iodine number of orange and bleached shellac, respectively, and 228 as the iodine number of rosin may give a slightly lower percentage of rosin, under some circumstances, than that which is actually present.

MOISTURE

Procedure

18. (*a*) *Orange Shellac and Dry-Bleached Shellac.*—Weigh a sample of 5 g. in a flat-bottomed dish about 4 in. in diameter and place the dish in a well-ventilated gas or electric oven for at least 6 hr. at 41 ± 2 C. Cool in a desiccator and weigh. Continue heating to constant weight.

NOTE.—Dry-bleached shellac is also termed "bone-dry," "kiln-dry," or "vac-dry" bleached shellac.

(*b*) *Hanks and Ground-Bleached Shellac.*—Weigh a 5-g. sample in a flat-bottomed dish about 4 in. in diameter and dry in a desiccator over H_2SO_4 for at least 12 hr. Place the dish in a well-ventilated gas or electric oven for at least 6 hr. at 41 ± 2 C., cool in a desiccator, and weigh. Continue heating to constant weight.

NOTE.—Average commercial orange shellac contains not more than 2 per cent of moisture. Average commercial regular dry-bleached and dry-refined bleached shellac contain not more than 6 per cent of moisture. Average commercial regular and refined bleached shellac in the form of hanks or bars, or ground bleached contain not more than 25 per cent of moisture.

WAX

Solvents Required

19. (*a*) 95 *per cent Alcohol.*—Specially denatured, 190-proof alcohol; Formula No. 1 or No. 30 of the U. S. Internal Revenue Bureau.

(*b*) *Chloroform.*—This solvent shall be redistilled before using as it must not leave a nonvolatile residue.

(*c*) *Filter Cel.*—The filter cel shall be extracted with the chloroform before using.

NOTE.—Average commercial orange and dry-bleached shellac will contain about 4 to 5.5 per cent of wax. Average commercial dry-bleached refined shellac will be practically free from wax.

Procedure

20. Dissolve 10 g. of shellac and 2.5 g. of Na_2CO_3 in 150 ml. of hot water. Make the solution in a 200-ml. tall-form beaker; immerse the beaker in a steam or boiling-water bath and stir until the shellac is in solution. Then cover with a watch glass and allow to remain in the bath for 2 to 3 hr. more without agitation. Remove the beaker from the bath and place it in cold water. The wax will come to the top where it will solidify as a layer or float as small hard particles according to the amount of wax present in the lac. Filter the solution through a 12-cm. folded filter paper or a Büchner funnel ($2\frac{1}{4}$ or $3\frac{1}{2}$ in. in inside diameter). Cover the bottom of the Büchner funnel with a disk of filter paper, mix 1 g. of filter cel with water, and pour onto the filter. Add 0.5 g. of filter cel to the solution, and filter with the aid of a vacuum. After washing out the soluble shellac, pour on a few milliliters of alcohol to facilitate drying. Dry at a temperature of 40 ± 2 C., wrap in a

filter paper, tie or bind with thin copper wire, and extract in a suitable continuous extraction apparatus with chloroform for 2 hr. Dry the wax at 105 C. to within 10 mg. of constant weight.

ORPIMENT IN ORANGE SHELLAC

Apparatus

21. (*a*) A jacketed glass funnel.

(*b*) A Kipp generator or its equivalent for a steady supply of hydrogen sulfide.

Special Solution Required

22. *Specially Denatured Alcohol* (either No. 1 or No. 30).

Procedure

23. (*a*) Weigh exactly 50 g. of the thoroughly rolled 25-mesh sample, transfer to a 500-ml. round bottom flask, add 450 ml. of specially denatured alcohol (either No. 1 or No. 30), and clamp the flask securely in a hot water bath. Allow to come to a gentle boil and complete solution of the shellac. Remove from bath and wait just long enough to permit settlement of the bulk of the insoluble matter, and filter through a 12.5-cm. No. 40 Whatman paper, or its equivalent, placed in either a funnel about which has been wound a coil of copper or tin tubing through which steam is passed to heat it, or preferably a jacketed glass funnel. For speedy filtration, it is absolutely imperative that both funnel and contents be kept hot, otherwise the wax will congeal and clog the paper. Keep a watch glass over the funnel when not pouring into it. Do not pour off too close from the insoluble residue, but wash flask and paper by four successive treatments with 50 ml. of the boiling alcohol, pouring the alcohol over all surfaces of the paper. Finally, wash the paper with 50 ml. of the boiling alcohol, and, when it has run through, remove the watch glass and allow steam to flow for a few minutes in order to drive out most of the alcohol. Change the receiver and pour portionwise, with steam on, 200 ml. of boiling carbon tetrachloride over the surface of the paper in order to remove residual wax which, if present, materially prolongs the subsequent digestion operation. Leave the cover off and allow steam to flow for a few minutes to drive out most of the solvent. Carefully remove the paper from the funnel and return it to its initial solution flask.

(*b*) To the flask and its contents add 25 ml. of arsenic-free HNO_3 (sp. gr. 1.42), place under a hood with good draft, and apply gentle heat, digesting for 20 min. (ample time for destruction of the paper and solution of the arsenic). Cool, add 25 ml. of arsenic-free H_2SO_4 (sp. gr. 1.84), return to hood, and heat gently at first until most of the nitrous fumes are off, after which increase heat and continue to evolution of sulfur trioxide fumes. If organic matter or charring is evident at this stage, cool the flask, rinse down sides sparingly with water, and add with caution 3 to 4 ml. of hydrogen peroxide (30 per cent). After initial violent gas evolution has ceased, heat again to sulfur trioxide fumes. If discoloration reappears, repeat the peroxide addition and the heating to fumes. Generally two such treatments suffice to produce a clear, carbon-free solution. When such a point is reached, transfer the solution to a 300-ml. Florence flask, rinsing thoroughly, so as to bring the final volume up to 125 to 150 ml. Boil this down rapidly to low bulk, just short of fuming, to insure complete (and necessary) removal of both HNO_3 and H_2O_2.

(*c*) Add 20 g. of either c.p. ferrous or ferrous ammonium sulfate to the contents of the flask and connect to an upright, bulb-type, glass condenser with an U-tube having about ¼-in. bore and

a distance of 8 in. between arms, using a two-hole rubber stopper in the flask. Through the other hole insert either a double-bulb safety tube or a small separatory funnel with a long slim tip. Bend the end of this tip into a short U so that the orifice points upward, and fit it into the flask so that it very nearly touches the bottom. Place a 250-ml. beaker containing 50 ml. of water on a support which is readily movable, so that the condenser tip is just immersed in the water during the entire distillation. Put into the beaker a glass tube connected with a supply of hydrogen sulfide, kept bubbling through at a good rate during the entire distillation. When the apparatus is securely connected, water flowing through the condenser, and hydrogen sulfide passing into the receiver, pour 50 ml. of arsenic-free HCl (sp. gr. 1.19) into the bulb of the safety tube and allow to flow into the flask. Apply heat gently at first and bring to a steady boil. The yellow orpiment precipitate will begin to show in the receiver as soon as distillation starts. Continue the distillation until bumping begins. Remove the flame, change the receiver, run another 50 ml. of HCl (sp. gr. 1.19) into the flask, and repeat the distillation, using the same procedure as before. With a properly prepared sample, practically all of the arsenic comes over in the first distillation, the second one serving mainly as a safeguard. When it is finished, lower the receiver, wash down the inside of the condenser and the outside of the condenser tip into the flask, and if any orpiment is observed on the walls of the condenser, wash sparingly with NH_4OH (sp. gr. 0.90), allowing it to drain and then rinsing again with water. Check the acidity of the contents of the beaker, adding more acid if necessary, and allow hydrogen sulfide to bubble through for a few minutes.

(*d*) In the meanwhile prepare a Gooch crucible with asbestos mat, ignite it, and cool in a desiccator. Weigh it as accurately as possible, since the precipitate in the final weighing may weigh 10 mg. or less. Filter contents of both beakers through the tared Gooch crucible, scrubbing thoroughly with a rubber policeman and then washing with hot water. After the water is through, wash with two 50-ml. portions of strong alcohol and then with two 50-ml. portions of boiling carbon disulfide (heating the carbon disulfide in a flask immersed in hot water, with flames out), regulating the suction in such a way as to retain each portion in the crucible for about 5 min. This treatment is very important, as it serves to remove any traces of sulfur which may have precipitated with the orpiment, and which if left in would materially affect the final result, particularly with a sample quite low in arsenic. Dry the Gooch crucible and contents in an oven at 105 to 110 C., a drying for 2 hr. generally being sufficient, cool in a desiccator, and weigh as accurately as possible. Calculate the gain in weight directly to percentage of As_2S_3 (orpiment).

(*e*) If there is any reason for believing that the reagents used are not arsenic-free, run blank determinations concurrently, using the same reagents in the same amounts, and deduct from the determination any arsenic thus found.

MATTER SOLUBLE IN WATER

Procedure

24. Weigh accurately 10 to 25 g. of the finely ground sample, and stir it thoroughly with 200 ml. of distilled water in a suitable-size flask or beaker. Cover with a watch glass and allow to stand at room temperature (approximately 21 C.) for 4 hr. (Note), stirring occasionally. Decant the water through a Büchner

funnel, 2½ or 3½ in. in inside diameter, the bottom of which is covered with a wet filter paper held tightly in position by suction. Wash the shellac and filter paper with at least 100 ml. of water. Evaporate the water and dry the extract to constant weight at 105 to 110 C. for 1 hr. or more. Cool, weigh, and calculate the percentage of matter soluble in water.

NOTE.—In the case of dry-bleached shellac, it is recommended that during the summer the vessel be kept at a temperature of 16.5 C. or less during the 4-hr. digestion period.

ASH

Procedure

25. Char a weighed quantity (3 to 5 g.) of the shellac and ignite at a low heat, not exceeding dull redness, until free from carbon. If a carbon-free ash can not be obtained in this manner, exhaust the charred mass with hot water, collect the insoluble residue on an ashless filter, burn the filter and contents until all the carbon is consumed. Add the filtrate, evaporate to dryness, and heat to dull redness. Cool in a desiccator and weigh.

COLOR

Procedure

26. Digest a weighed portion of the shellac with twice its weight of cold 95 per cent alcohol, shaking at intervals until the shellac is entirely "cut" and proceed in accordance with Section 29 by the particular method agreed upon by the purchaser and the seller.

SHELLAC VARNISH

Temperature of Testing

27. All tests, unless otherwise specified, shall be made at room temperature between 21 and 32 C. (70 and 90 F.).

Sampling

28. The sample of shellac varnish shall be thoroughly agitated in the container immediately before portions are removed for the various tests, and the unused portion shall be kept in a tightly stoppered glass container, in a cool, dark place.

COLOR

Procedure

29. Compare the color of the sample with the color of the shellac varnish agreed upon by the purchaser and the seller, by either of the following methods:

(*a*) *Comparison of Solutions.*—Shake both shellac varnishes well and then place equal portions of each in separate clear-glass tubes of the same diameter. Make the color comparison by placing the tubes together and comparing the colors.

(*b*) *Comparison of Films.*—Shake the shellac varnishes well and then allow them to stand for 30 min. Flow approximately equal portions of the two shellac varnishes on separate milk-glass or porcelain plates and allow the films to dry in a vertical position. Make the color comparison upon the dried films.

NONVOLATILE MATTER

Procedure

30. Pour a portion of the sample of shellac varnish into a stoppered bottle or weighing pipette and weigh. Transfer not more than 1.5 g. of the sample to a weighed metal dish. Weigh the container again, and calculate by difference the exact weight transferred to the dish. The tared metal dish shall have a flat bottom about 8 cm. in diameter and shall contain about 10 g. of prepared sea sand (Note). A friction-top can cover will serve as a satisfactory dish. Add approximately 2 g. of alcohol

to the contents of the dish and mix thoroughly with a glass rod. Heat the dish and its contents for 3 hr. in an oven maintained at 105 ± 2 C. Place in a desiccator, cool, and weigh. From the weight of the residue and sample calculate the percentage of nonvolatile matter.

NOTE.—The sand shall have previously been prepared by digesting with hot HCl for 1 hr., washing to remove all acid and soluble impurities, igniting, and then cooling in a desiccator.

WAX

Procedure

31. Weigh a quantity of the shellac varnish that will contain approximately 10 g. of nonvolatile matter, and place in a 200-ml. tall-form beaker, to which add 2.5 g. of Na_2CO_3 and 150 ml. of hot water. Immerse the beaker in a steam or boiling-water bath and stir until the shellac is in solution. Then cover with a watch glass and complete the determination in accordance with the procedure described in Section 20.

INSOLUBLE MATTER

Procedure

32. Determine insoluble matter in accordance with either method *A* or *B* as described in Sections 4 to 11. The quantity of shellac varnish taken shall correspond, as closely as practicable, to 5 g. of nonvolatile matter found in the sample.

IODINE NUMBER

Procedure

33. Pour the shellac varnish on a perfectly clean glass plate, 6 by 10 in., and place the plate in a nearly vertical position in a well ventilated room at 20 to 30 C. (70 to 90 F.) for at least 1 hr. to allow the excess varnish to drain off. Then place the plate horizontally in an air bath overnight at a temperature of 43 ± 5 C., cool, and scrape the dry shellac from the plate with a razor blade, discarding the thick edges. Weigh accurately a 0.2 ± 0.005 g. sample of the dry shellac, and transfer to 250-ml. glass-stoppered bottle. In the case of badly adulterated samples, use a 0.1 ± 0.005 g. sample. Determine the iodine number in accordance with the procedure for dry shellac described in Sections 12 to 14.

PURITY

Qualitative Tests

34. Make qualitative tests for rosin upon the residue from shellac varnish evaporated at 75 to 80 C., and for copal upon the filtrate of shellac varnish filtered through dry filter paper in accordance with Sections 15 and 16.

Estimation of Adulterant

35. If the qualitative test shows the presence of rosin but no copal or the presence of copal but no rosin, the percentage of rosin or copal, respectively, may be estimated from the iodine number as described in Section 17.

APPROXIMATE DRYING TIME

Procedure

36. Dilute the sample of shellac varnish with alcohol until it contains only 36 per cent or less of nonvolatile matter. Pour the shellac varnish on a perfectly clean glass plate, at least 2 by 4 in., and place the plate in a nearly vertical position in a well ventilated room but not in the direct rays of the sun. The temperature of the room should be between 20 and 30 C. (70 and 90 F.) (Note). Test the varnish film at points not less than 2.5 cm. from the top and side edges of the film by touching lightly with the finger. The shellac varnish shall be considered to have "set to touch" when gentle pressure of the finger shows a tacky condition but none of the varnish adheres to the finger. The shellac varnish shall be considered

to have "dried hard" when the pressure that can be exerted on the film by the thumb and finger, when the film is placed between them, does not move the film or leave a mark that remains noticeable after the spot is lightly polished.

NOTE.—In cases of dispute, this test shall be made in a well ventilated room maintained at a temperature of 20 C. (70 F.) and a relative humidity of 65 per cent.

NATURE OF VOLATILE MATTER

Procedure

37. Information concerning the volatile portion of a shellac varnish may be obtained by distilling an accurately weighed 50 to 100-ml. sample of shellac varnish in a 250 or 300-ml. wide-neck Erlenmeyer flask, which is fitted with a two-hole rubber stopper to take a thermometer and a short bent glass tube. Connect this bent glass tube by means of rubber tubing to a 12-in. Liebig condenser set downward for distillation (the diameter of the inner tube of the condenser shall be equal to the diameter of the ends of the condenser and the diameter of the bent glass tube). Add a quantity of glass beads and immerse the flask deeply in an oil or wax bath[12] heated to 90 C. Gradually raise the temperature during the first hour to 120 C. and continue the distillation at this temperature for 2 hr. A specific gravity determination on the distillate will give the approximate strength of the solvent. A portion of the final distillate may be diluted with water to ascertain whether solvents other than alcohol, such as naphtha or benzol, have been added. These will separate on dilution and the layer formed may be measured.

[12] Fisher's bath wax is recommended.

WEIGHT PER GALLON

Procedure

38. Determine the specific gravity of shellac varnish at 20 C. by any convenient method that is accurate to the third significant figure (Note). A convenient method of determining specific gravity is to weigh a clean, dry 100-ml. volumetric flask calibrated at 20 C., fill to the mark with the shellac varnish at 20 C., and again weigh. The increase in weight divided by the capacity of the flask in grams of water is the specific gravity at 20 C. The specific gravity multiplied by 8.33 gives the weight in pounds per U. S. gallon.

NOTE.—The hydrometer does not give reliable or accurate indication of the specific gravity of shellac varnish.

Standard Methods of

TESTING OLEORESINOUS VARNISHES[1]

A.S.T.M. Designation: D 154 – 43

ADOPTED, 1924; REVISED, 1925, 1928, 1938, 1943.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 154; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods cover procedures for testing oleoresinous varnishes.

General Requirements for Tests

2. (*a*) All tests shall be made at room temperature between 21 and 32 C. (70 and 90 F.).

(*b*) All tests shall be made in diffused light (not in direct sunlight).

(*c*) When a can of varnish has been opened and part of its contents has been used, the remainder shall immediately be placed in airtight containers which the varnish almost entirely fills, leaving not more than 2 per cent of air space.

APPEARANCE

Procedure

3. Some of the thoroughly mixed sample shall be poured into a clear glass bottle or test tube, 1.5 to 2.0 cm. ($\frac{5}{8}$ to $\frac{13}{16}$ in.) in diameter to a depth of at least 2.5 cm. The varnish shall then be examined by transmitted light and shall be clear and transparent.

COLOR

Color Reference Standards

4. (*a*) Reference standard color solutions shall be prepared by dissolving 1, 2, 3, 4, 5, and 6 g., respectively, of pure powdered potassium dichromate in 100 ml. of pure H_2SO_4 (sp. gr. 1.84). Gentle heat may be applied, if necessary, to effect solution of the dichromate.

(*b*) Since the potassium dichromate - sulfuric acid solutions must be freshly made for this color comparison, it is frequently more convenient to compare samples with a series of permanently sealed tubes of varnish which have been previously found to be lighter in color than the reference standard solutions.

NOTE.—Stabilized caramel solutions or other permanent colored liquids may be used as secondary reference standards.

Procedure

5. The reference standard color solutions and a sample of the varnish to be tested shall each be poured into separate thin-walled glass tubes 1.5 to 2.0 cm. ($\frac{5}{8}$

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these methods were published as tentative from 1923 to 1924.

to $\frac{13}{16}$ in.) in diameter to a depth of not less than 2.5 cm. The color comparison shall be made by placing the tubes close together and looking through them by transmitted light. If permanently sealed tubes of varnish are used for the color comparison and the sample of the varnish being tested is found to be darker than a standard tube of varnish, final comparison shall be made with freshly prepared potassium dichromate - sulfuric acid solutions.

Report

6. The color of the varnish shall be reported in terms of the reference standard (calling the standards No. 1, No. 2, No. 3, etc.) which it is equal to or lighter than in color.

NONVOLATILE MATTER

Procedure

7. A portion of the sample of the varnish shall be poured into a stoppered bottle or weighing pipette and weighed. About 1.5 g. of the sample shall be transferred to a weighed, flat-bottom metal dish (Note) about 8 cm. in diameter (a friction-top can cover is satisfactory). The container shall be weighed again, and the exact weight of the portion of the sample transferred to the weighed dish shall be calculated by difference. The dish with its contents shall be heated for 3 hr. in an oven maintained at 105 to 110 C. (221 to 230 F.). It shall then be weighed after cooling.

NOTE.—A pyrex or similar heat-resistant glass Petri dish 100 mm. in diameter by 10 mm. in depth may be used in place of the flat-bottom metal dish.

Calculation

8. The ratio of the weight of the residue to that of the sample, expressed as a percentage, shall be taken as the percentage of nonvolatile matter in the varnish.

ELASTICITY OR TOUGHNESS

9. The elasticity or toughness of the varnish shall be determined by proportionately reducing its elasticity or toughness by the addition of a standard solution of Run Kauri gum in pure spirits of turpentine as described in method *A*, or by proportionately increasing the elasticity or toughness of the varnish by means of addition of linseed oil as described in method *B*.

Method A. Run Kauri Reduction

Preparation of Standard Run Kauri Solution

10. (*a*) A distillation flask, a water-cooled condenser, and a tared receiver shall be arranged on a balance. Clear, bright, hard pieces of Kauri gum broken to the size of a pea shall be placed in the flask to about one third of its capacity. The gum shall be carefully melted and distilled until 25 per cent by weight is collected in the tared receiver. At the end of the distillation, the thermometer in the distillation flask, with the bulb at the level of the discharging end of the flask, should register about 316 C. (600 F.). The residue shall be poured into a clean pan, and when cold it shall be broken into small pieces.

(*b*) A quantity of the small broken pieces of Run Kauri, together with twice its weight of freshly redistilled spirits of turpentine, using only that portion distilling between 153 and 170 C. (308 and 338 F.), shall be placed in a carefully tared beaker and dissolved by heating to a temperature of about 149 C. (300 F.). It shall then be brought to correct weight, when cooled, by the addition of the amount of redistilled spirits of turpentine necessary to replace the loss by evaporation during the dissolving of the gum.

Test Panels

11. Test panels shall be cut from bright tin plate weighing not more than 25 nor less than 19 g. per sq. dm. (0.51 to 0.39 lb. per sq. ft.). It is important that the tin plate shall be within the limits prescribed. The panel shall be about 7.5 by 13 cm. (3 by 5 in.) and shall be thoroughly cleaned with benzol immediately before using.

NOTE.—Commercial No. 31 gage bright tin plate should weigh about 0.44 lb. per sq. ft. It is important that the rags used in wiping the panels are clean.

Procedure

12. (*a*) Having carefully determined the nonvolatile content of the varnish in accordance with Sections 7 and 8, 100 parts of the varnish by weight shall be taken and to it shall be added an amount of the standard Run Kauri solution equivalent to 50 per cent by weight of the nonvolatile matter in the varnish and mixed thoroughly.

NOTE.—The 50 per cent standard Run Kauri reduction is given to illustrate the method. Any other percentage of standard Run Kauri reduction may be used, depending on what is required of the particular sample being tested, see Section 13.

(*b*) The varnish shall be flowed upon one of the tin panels described in Section 11 and the panel shall be permitted to stand in a nearly vertical position at room temperature for nearly 1 hr. The panel shall then be placed in a horizontal position in a properly ventilated oven and baked for 5 hr. from 95 to 100 C. (203 to 212 F.). The panel shall then be removed from the oven and permitted to cool at room temperature (preferably 24 C. (75 F.)) for 15 min.

(*c*) *Bending the Test Panel.*—The panel shall be placed with the varnished side uppermost over a 3-mm. ($\frac{1}{8}$-in.) rod, held firmly by suitable supports, at a point equidistant from the top and bottom edges of the panel and bent double rapidly. The varnish shall show no cracking whatsoever at the point of bending. For accurate results the bending of the panel should always be done at 24 C. (75 F.), since a lowering of the temperature will lower the percentage of reduction that the varnish will stand without cracking, while an increase in temperature increases the percentage of reduction that the varnish will withstand.

Report

13. (*a*) Varnishes which do not show cracks under this test shall be reported as passing a 50 per cent reduction, while those that do crack shall be reported as not passing a 50 per cent reduction.

(*b*) The varnishes which have not cracked shall be tested again, changing the amount of reduction to 60 per cent, and, if they pass this percentage of reduction, they shall be tested with a 70 per cent reduction. In a similar manner, varnishes which have cracked at 50 per cent shall be tested again, using reductions of 40 and 30 per cent. In this way the limits shall be determined within 10 per cent at which a varnish passes one percentage of reduction and does not pass the next. For example, varnishes may be reported as passing 40 per cent, and breaking at 50 per cent.

NOTE.—It is suggested that a 20-g. sample of varnish shall be sufficient for each reduction. If the nonvolatile content of the varnish should be 48.6 per cent, then 4.86 g. of standard Run Kauri solution should be added to the 20 g. of varnish and a 50 per cent reduction will be obtained.

Method B. Addition of Linseed Oil

Scope

14. This method is applicable to varnishes which are less elastic or tougher than zero Kauri reduction. It is identical with that described in method *A* except that the 33$\frac{1}{3}$ per cent

solution of Run Kauri in turpentine is replaced by a 66⅔ per cent solution of heat bodied linseed oil for proportionately increasing the elasticity of the varnish under test.

Preparation of Standard Bodied Oil Solution

15. (*a*) A high grade of alkali-refined linseed oil having an acid number less than 1.0 shall be heated in an open kettle at a temperature of 300 ± 5 C. until the viscosity of the oil after cooling shall be between 6 and 10 poises at 25 C. (77 F.).

(*b*) A quantity of the heat bodied linseed oil shall be standardized by reducing it with one-half its weight of pure redistilled turpentine, using only that portion of turpentine distilling between 153 and 170 C. (308 and 338 F.).

Procedure

16. The addition of the standard bodied oil solution to the varnish and the flowing on, baking, and bending of the test panel shall be performed exactly as described in Sections 12 and 13.

Report

17. In reporting results, the minimum percentage of the oil solution that must be added to the varnish, based on its nonvolatile content, so that the final mixture when flowed on a test panel and baked does not crack on the subsequent bending over a 3-mm. rod shall be reported.

FLASH POINT

Procedure

18. The flash point shall be determined in accordance with the Standard Method of Test for Flash Point by Means of the Tag Closed Tester (A.S.T.M. Designation: D 56) of the American Society for Testing Materials.[3]

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

VISCOSITY

Procedure

19. Viscosity shall be determined by comparison at 25 C. (77 F.) with secondary standards whose viscosity expressed in poises has been accurately determined at that temperature.

NOTE.—Gardner bubble viscosimeter tubes[4] may be used.

WATER TEST

Procedure

20. The varnish shall be poured on one of the tin panels described in Section 11, and allowed to drain in a nearly vertical position and dry for 48 hr. The panel shall then be placed in a beaker containing about 7 cm. (2.5 in.) of distilled water at room temperature, immersing the end of the panel which was uppermost during the drying, and allowed to remain in the water for 18 hr. The panel shall then be removed from the water, wiped carefully, and allowed to dry out at room temperature. The time required for whitening, if any, to disappear, shall be noted. Blooming, which sometimes occurs on immersion, is considered as a degree of whitening.

Report

21. The results of the water test shall be reported as follows:

(*a*) Not visibly affected,

(*b*) Whitening disappears within 20 min.,

(*c*) Whitening does not disappear in 20 min., but does disappear within 2 hr.,

(*d*) Whitening does not disappear within 2 hr., but does disappear within 24 hr., and

(*e*) Whitening does not disappear within 24 hr.

[4] "Physical and Chemical Examination of Paints, Varnishes, Lacquers, and Colors," National Paint, Varnish, and Lacquer Assn., Ninth edition, p. 216 (1939).

SKINNING TEST OF VARNISH IN CLOSED CONTAINERS

Container

22. An 8-oz. wide mouth glass jar 4½ in. in height and 2 in. in diameter shall be used as the container.[5]

Procedure

23. A 6-oz. sample of the varnish shall be measured into the glass jar container by means of a graduate. The cover shall be screwed on tightly, and the jar inverted momentarily. The jar shall then be placed in an upright position in the dark (placing it under a box or in a drawer is satisfactory) and the varnish shall be examined for skinning at specified time intervals.

ALKALI RESISTANCE TEST

Procedure

24. A 1 by 6-in. test tube shall be thoroughly cleaned in benzol. The tube shall then be dipped into the sample of varnish and the mouth of the tube inverted downward immediately and the varnish allowed to dry for 48 hr. in an atmosphere free from dust, drafts, products of combustion, or laboratory fumes. The temperature of the room during the drying period shall be 65 to 78 F. (19 to 25 C.). The varnish-coated tube shall then be suspended in a solution of sodium hydroxide (3 per cent) maintained at 70 ± 2 F. (21 ± 1.1 C.). The tube shall be removed after desired periods of time, such as 8, 16, 24, and 48 hr., and the varnish examined for whitening, blistering, or removal. Before making the examination, the varnished tube shall be rinsed under a gentle stream of water and allowed to air dry for 30 min.

[5] Jar No. 5107 C. T. with 12S P. and O. Cap, of the Hazel-Atlas Glass Co., Wheeling, W. Va., has been found suitable.

ACID NUMBER

Solutions Required

25. (*a*) *Alcoholic Oxalic Acid Solution (0.1 N).*—Dissolve 6.3 g. of c.p. oxalic acid ($H_2C_2O_4 \cdot H_2O$) in sufficient 95 per cent ethyl alcohol to make a liter.

(*b*) *Alcoholic Potassium Hydroxide (0.1 N).*—Dissolve approximately 6.6 g. of c.p. alcohol-refined potassium hydroxide (85.86 per cent KOH) in sufficient 95 per cent ethyl alcohol to make a liter, and standardize in the usual manner.

(*c*) *Neutral Solvent Mixture.*—Mix 75 per cent of ethyl alcohol (95 per cent) and 25 per cent of c.p. benzene (benzol, C_6H_6).

(*d*) *Phenolphthalein Indicator.*—Dissolve 10 g. of phenolphthalein indicator in 1 liter of ethyl alcohol (95 per cent). Use 1 ml. for titration.

Procedure

26. (*a*) A 3 to 5-g. sample of the varnish shall be weighed into a 500-ml. glass-stoppered Erlenmeyer flask, 75 ml. of the solvent mixture added and warmed slightly to aid solution. A definite accurately measured quantity (10 to 25 ml.) of the standard 0.1 *N* oxalic acid solution shall then be added. The mixture shall be refluxed on a hot plate (using a reflux condenser) for about 1 hr., or until the soaps present have been decomposed. The solution shall then be removed and titrated while hot with the standard 0.1 *N* potassium hydroxide solution using phenolphthalein indicator. As the end point is approached, very vigorous shaking will be required. Titrating while hot increases the speed of titration and results in a sharper end point. A pink coloration which persists for several minutes shall be taken as the end point.

(*b*) A blank containing the same quantity of the standard 0.1 *N* oxalic

acid solution should be run using the same procedure as for the sample.

Calculation

27. The acid number is the weight in milligrams of potassium hydroxide required to neutralize a 1-g. sample of the varnish, calculated as follows:

$$\text{Acid number} = \frac{(a - b)c}{w}$$

where:

a = milliliters of potassium hydroxide required for the sample,
b = milliliters of potassium hydroxide required for the blank,
c = milligrams of potassium hydroxide per milliliter (value of standard potassium hydroxide solution), and
w = weight of the sample in grams.

Standard Method of Test for

REACTIVITY OF PAINT LIQUIDS[1]

A.S.T.M. Designation: D 479 - 40

ADOPTED, 1940.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 479; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test is intended for determining the reactivity between paint liquids and zinc oxide.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Balance.*—Balance or scale sensitive to 1 g.

(*b*) *Containers.*—Friction-top cans.

(*c*) *Spatulas.*—Spatulas or suitable paddles.

(*d*) *Thermometer.*—Thermometer graduated from 0 to 100 C. and accurate to 0.5 C.

(*e*) *Paint Mixing Equipment* (Optional).—Laboratory paint mixer or stirrer.

(*f*) *Consistency Measuring Apparatus* (Optional).—Mobilometer, viscosimeter, plastometer, or viscosity cup.

Materials

3. The following materials will be required:

(*a*) *Paint Liquid.*—Varnishes, resin solutions, or other liquids used in the manufacture of paints, enamels, and lacquers are considered as paint liquids. If the viscosity of the liquid to be tested is such that dilution is necessary, the liquid reduced in a manner mutually agreed upon by the purchaser and the seller shall be used in the test.

(*b*) *Zinc Oxide.*—A standard Green Seal zinc oxide or other zinc oxide mutually agreed upon by the purchaser and the seller.

Procedure

4. The zinc oxide and paint liquid in a ratio of 35 to 65 by weight, respectively, shall be mixed to a uniform paint. Usually 210 g. of zinc oxide and 390 g. of paint liquid are ample. The zinc oxide and paint liquid shall be mixed by alternately adding portions of the pigment and liquid. The additions shall be regulated so that a fairly stiff paste is produced. After all the pigment and sufficient liquid have been added to produce a paste, the paste shall be reduced to a paint by gradually working in the remainder of the liquid. The total mix-

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, this method was published as tentative from 1938 to 1940.

ing operation shall not take longer than 30 min. The mixing may be done in a suitable container by hand with a stiff spatula or a paddle, or with a suitable mechanical mixer. The temperature of the paint shall then be brought to 25 ± 0.5 C. and its consistency determined by a method mutually agreed upon by the purchaser and the seller, which may include visual inspection with a spatula, or any of the usual instruments such as the Gardner mobilometer or the Stormer viscosimeter. Limits for the consistency of the fresh paint shall be agreed upon by the purchaser and the seller. The paint shall then be transferred to a friction-top can and stored under normal room conditions. The can shall be filled to within at least ¼ in. of the top and the lid tightly inserted. After 24-hr. storage and again after 5-day storage (Note) the paint shall be thoroughly mixed, brought to 25 ± 0.5 C. and the consistency determined, using the same procedure as used in determining the consistency of the fresh paint.

NOTE.—Preliminary indications of the reactivity of the liquid are obtained after 24-hr. storage, but final decision should be made following the consistency determination after the 5-day storage. By mutual agreement between the purchaser and the seller, any specific period of storage may be used.

Grading

5. The paint liquid shall be graded as nonreactive, slightly reactive, moderately reactive, or reactive, in accordance with the following:

(*a*) *Nonreactive.*—If the consistency of the aged paint is the same or less than that of the fresh paint, the liquid shall be graded as nonreactive.

(*b*) *Slightly Reactive.*—If on aging the consistency increases slightly but not sufficiently to be of practical significance, the liquid shall be graded as slightly reactive.

(*c*) *Moderately Reactive.*—If on aging the consistency definitely increases but the product would still be usable, the liquid shall be graded as moderately reactive.

(*d*) *Reactive.*—If on aging the paint bodies objectionably, livers, or gells, the liquid shall be graded as reactive. The purchaser and the seller shall agree on the maximum increase in consistency that will be permissible.

Standard Methods of

TESTING LIQUID DRIERS[1]

A.S.T.M. Designation: D 564 – 43

ADOPTED, 1943.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 564; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods cover the test procedures to be applied to liquid driers for use in paints and varnishes.

Conditioning

2. The driers shall be held at a temperature between 70 and 80 F. (21 and 27 C.) for at least 18 hr. prior to examination.

PHYSICAL TESTS

Appearance

3. Examine the sample and note any separation, either of sediment or of suspended matter.

Sediment or Suspended Matter

4. Weigh a sample of convenient size, approximately 1 to 5 g. Filter through a Gooch crucible prepared in the usual manner, wash with turpentine, and dry at 49 C. to constant weight.

Color

5. Determine the color of the sample as received. Then add 1 volume of the sample to 9 volumes of raw linseed oil and determine the color. Determine the color by comparison with one of the following reference color standards: potassium dichromate – sulfuric acid, Gardner-Holdt, or Hellige-Klett.

Nonvolatile Matter

6. Weigh by difference 1.5 g. of the sample into the cover from a friction-top can. Heat at 105 to 110 C. for 3 hr., cool, and weigh.

Miscibility with Oil

7. Mix 1 volume of the sample with 19 volumes of raw linseed oil. Record any signs of separation or clouding. Then observe the mixture at 1-hr. intervals for 3 hr., and again after 24 hr.

NOTE.—In the case of disagreement between the seller and the purchaser, the test for miscibility with oil shall be made at 25 ± 1 C.

Drying Power

8. Blend 1 volume of the sample with 9 volumes of raw linseed oil, and

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products

[2] Prior to adoption as standard, these methods were published as tentative from 1940 to 1943, being revised in 1941 and 1943.

flow on a clean glass plate. Place the plate in a vertical position in air at 20 to 30 C. and a relative humidity of 40 to 60 per cent. Record the time required for the film to set to touch, which is the time when the oil does not stick to the finger or the surface is not marred when the finger is lightly drawn across it.

CHEMICAL ANALYSIS

Lead

Procedure

9. Weigh by difference (using a dropping bottle) about 5 g. of the sample into a 400-ml. beaker with cover (or use a homeopathic vial of the size of a Gardner-Holdt tube in which to weigh the sample and transfer it to the beaker). Heat on a hot plate until the solvent is removed. Add about 5 ml. of HNO_3 (sp. gr. 1.42) and 40 to 50 ml. of H_2SO_4 (1:1). Evaporate to dense white fumes of SO_3 and continue the evaporation until about 5 ml. of acid remain. If the solution turns dark, add very carefully, by means of a dropping bottle, a few drops of HNO_3 (sp. gr. 1.42). Repeat this process until the solution remains clear after evaporation to white fumes. Allow to cool, then add carefully a few milliliters of H_2O_2 (30 per cent). Boil for 1 min., cool, add carefully about 100 ml. of distilled water, and put the solution on a hot plate for a few minutes to allow crystals to coagulate. Filter through a Gooch crucible, previously prepared and heated to constant weight. Wash with H_2SO_4 (0.5 per cent), then with a little alcohol. Place the crucible in a muffle furnace and heat for 15 min. at a dull red heat (not over 600 C.), cool, and weigh as $PbSO_4$. Calculate the percentage of lead as follows:

$$\text{Lead, per cent} = \frac{\text{wt. of } PbSO_4 \times 0.683}{\text{wt. of sample}} \times 100$$

Manganese

Reagents

10. (*a*) *Standard Vanadyl Sulfate.*—Dissolve 12 g. of ammonium metavanadate (chloride-free) in 400 ml. of hot water. Add 10 g. of $NaHSO_3$ followed by 20 ml. of H_2SO_4 (sp. gr. 1.84). A clear blue solution should result. If a precipitate forms, digest until dissolved. Concentrate by brisk boiling to remove the last traces of excess SO_2, cool, and dilute to 1 liter. Standardize against 0.1 *N* $KMnO_4$ as follows: In the standardization it is preferable to measure the permanganate into a 500-ml. flask, dilute to 300 ml. with water, and acidify with 7.5 ml. each of H_2SO_4 (sp. gr. 1.84) and H_3PO_4 (85 per cent). Titrate rapidly with the vanadium solution until the permanganate color changes to a faint pink color. Continue the titration drop by drop, allowing about 10 sec. between drops, until a clear yellow develops. Reserve the solution for use as a reference color standard, because many shades of yellow are possible and therefore a reference standard should always be used. Reduction of the measured permanganate followed by reoxidation according to the procedure does not yield a sufficiently permanent end point to serve as a color reference, because of slow reoxidation of manganese due to the presence of silver and persulfate. The standard vanadium solution is quite stable toward light and air.

(*b*) *Standard Potassium Permanganate (0.1 N).*—Dissolve 3.25 g. of $KMnO_4$ in 1000 ml. of water. Standardize against the National Bureau of Standards' standard sample 40*c* of sodium oxalate.

Procedure

11. (*a*) Weigh 10 g. of the sample into a 200-ml. tall-form beaker. Heat the

sample to a temperature just high enough to evaporate most of the volatile thinner, avoiding temperatures above 200 C., before proceeding with the double decomposition described in Paragraphs (*b*) and (*c*).

(*b*) Add 100 ml. of HCl (1:4) and 5 g. of beeswax, then warm just below the boiling point, while stirring occasionally to aid double decomposition. The reaction starts immediately, but about 1 hr. is usually required to completely liberate the fatty acids or whatever other organic acid may have been used in preparing the drier. The liberated acids rise to the surface as a clear yellow oily layer, while undecomposed particles settle to the bottom. The wax serves to absorb the liberated organic acids, and the mixture, upon cooling, solidifies and thereby enables separation by filtration. When decomposition is complete, thorough agitation is advisable so that all of the more or less emulsified organic acids may be extracted by the molten wax. Cool to room temperature, remove the solidified wax, and wash with a stream of water. Filter off the remaining particles of wax and wash to dissolve lead chloride (if much lead was originally present) and other drier metals. Make the filtrate distinctly ammoniacal, and precipitate the metals with *colorless* ammonium monosulfide. Digest to coagulate the insoluble sulfides, filter, and wash with diluted *colorless* ammonium monosulfide. Reserve the filtrate for a calcium determination, if desired, and reserve the sulfide precipitate for determining the other metals.

(*c*) Transfer the filter paper from the above separation, containing the sulfide precipitate, to a 250-ml. beaker, and add 10 ml. of HNO_3 (sp. gr. 1.42) and 5 ml. of H_2SO_4 (sp. gr. 1.84). Cover the beaker and slowly evaporate to fumes. Usually this treatment is sufficient to oxidize the organic matter, but if charring occurs after strong fuming, add a few milliliters more of HNO_3 and again evaporate to fumes. Wash the cover and sides of the beaker with water and evaporate to fumes. Repeat to insure complete removal of HNO_3. Add 100 ml. of water, and digest on a hot plate to insure complete solution of the anhydrous sulfates of other metals that may be present. Let stand at room temperature several hours. Filter, and use the filtrate for the determination of manganese as described in either method *A* or *B* (Paragraph (*d*) or (*e*)).

(*d*) *Method A.*—To the filtrate, add 7.5 ml. of H_3PO_4 (85 per cent), and dilute to about 150 ml. Add 10 ml. of 0.1 *N* $AgNO_3$ for each 0.05 g. of manganese present, and 3 g. of ammonium persulfate dissolved in about 15 ml. of water. Heat to just below the boiling point until a permanganate color fully develops. During the heating, various color changes may be observed: pale pink, brown, red, and permanganate color. If a brown precipitate is formed that does not clear by the time the boiling point is reached, too much manganese is present; if the color does not change beyond red, too much acid is present. If the brown precipitate forms, reduce the solution with H_2SO_3, dilute to volume, and aliquot; if the red color does not change to permanganate color, repeat the test using another sample. It should be remembered that 5 per cent of H_2SO_4 and 5 per cent of H_3PO_4 should not be exceeded. It should also be remembered that prolonged heating may destroy all the excess persulfate and thereby cause low results. Remove the solution from the heat source just as soon as the permanganate color fully develops, add 150 ml. of water, cool below 20 C., and titrate with the vanadium solution, as described for standardization (Section

10 (*a*)), to the same yellow color as obtained in the standardization.

(*e*) *Method B (Alternative).*—Cool the filtrate in iced water until the temperature of the solution reaches 15 C. or slightly less. Now add about 2 g. of sodium bismuthate, allowing the beaker and contents to remain in the iced water for at least 10 min. Add 50 ml. of diluted HNO_3 (3:97), filter through an asbestos pad, and wash the residue with cool diluted HNO_3 (3:97) until free of permanganic acid. Add enough standard $FeSO_4$ solution to reduce all of the permanganic acid and to require approximately 10 ml. of $KMnO_4$ in the back-titration. Stir thoroughly until the permanganic acid is decolorized. Titrate at once with 0.1 *N* $KMnO_4$ (Section 10 (*b*)) to the usual end point.

Carry a blank determination through all steps of the procedure, add the same amount of $FeSO_4$ as used for the sample, and titrate with $KMnO_4$. The difference between the titrations of the sample and blank represents the $KMnO_4$ equivalent of the permanganic acid formed in the test. From the manganese titer of the $KMnO_4$ solution, calculate the amount of manganese present.

Cobalt (Gravimetric Method)

Reagent

12. *Nitroso-β-Naphthol Solution.*—Dissolve 2 g. of nitroso-β-naphthol[3] in 50 ml. of hot glacial acetic acid. Add 50 ml. of hot water and filter.

NOTE.—This reagent will not precipitate nickel or manganese.

Procedure

13. (*a*) Weigh by difference (using a dropping bottle) 5 g. of the sample into a large crucible. Ash in the usual manner. After complete ashing, add 5 to 10 g. of potassium pyrosulfate ($K_2S_2O_7$) and heat until bubbling ceases, then with the full flame of the burner. When the molten mass attains a cherry-red heat, swirl the crucible gently, by means of crucible tongs, until the contents are completely fused and homogeneous.

(*b*) Cool the crucible to room temperature and extract the fused mass with 50 to 100 ml. of hot diluted H_2SO_4 (1:99). Filter through a fine filter paper to remove insoluble sulfates, carbon, etc., and dilute to 500 ml. in a volumetric flask.

(*c*) To an aliquot containing approximately 50 mg. of metallic cobalt, add 10 drops of hydrogen peroxide (30 per cent), 1 or 2 drops of phenolphthalein indicator, and then enough 0.5 *N* NaOH to precipitate the cobalt completely and show an alkaline reaction with the indicator present. Add 20 ml. of glacial acetic acid, and heat gently; then add 1 or 2 drops of hydrogen peroxide (30 per cent), and continue heating until the precipitate of $Co(OH)_3$ is dissolved. Filter at this point, even though no insoluble matter is apparent. Dilute to 200 to 250 ml. with water, heat to gentle boiling, and add 15 ml. of a freshly prepared, hot solution of nitroso-β-naphthol. Stir until the precipitate coagulates, and allow to stand for 2 hr. at 60 C. Filter through paper, preferably supported by a platinum cone, and wash with hot diluted HCl (1:19), followed by hot water to wash out the free acid. Apply gentle suction, and suck as dry as possible. Carefully ash in a tared porcelain crucible. Ignite for 1 hr. at 750 to 850 C., cool, and weigh as Co_3O_4.

$$\text{Cobalt, per cent} = \frac{\text{wt. of } Co_3O_4 \times 73.42}{\text{wt. of sample}}$$

Cobalt (Electrolytic Method)

Apparatus

14. The apparatus shall consist of suitable equipment for electrolytic anal-

[3] Merck's reagent quality, approved for cobalt determination, has been found satisfactory for this purpose.

ysis, of the type commercially available.[4]

Procedure

15. (*a*) Weigh 10 g. of the sample into a 200-ml. beaker. Heat the sample to a temperature just high enough to evaporate most of the volatile thinner, avoiding temperatures above 200 C.

(*b*) Add 100 ml. of H_2SO_4 (1:9) and 5 g. of beeswax, then warm just below the boiling point, while stirring occasionally to aid double decomposition. The reaction starts immediately, but about 1 hr. is usually required to completely liberate the fatty acids or whatever other organic acid may have been used in preparing the drier. Cool, filter, and wash 5 times with cool diluted H_2SO_4 (5 per cent). Dry and ash the beeswax and examine it for cobalt. Evaporate the filtrate to fumes. Oxidize the small amount of organic matter still retained by the solution by adding a few milliliters of HNO_3 (sp. gr. 1.42) and again evaporating to fumes. If charring occurs after strong fuming, repeat the fuming with HNO_3. Add an equal volume of water and again evaporate to fumes. Repeat to insure complete removal of HNO_3. Dilute to about 150 ml. and warm to dissolve the anhydrous cobalt sulfate. Add 2 g. of sodium bisulfite; 60 ml. of NH_4OH, and about 0.5 ml. of hydrogen peroxide (30 per cent), and stir until solution is complete. Dilute to 225 ml. with water; then plate out the cobalt on a tared platinum electrode, using a current of 0.3 amp. per sq. dm. When the electrolyte becomes colorless, transfer about 0.5 ml. to a small porcelain crucible, add a few crystals of phenylthiohydantoic acid, and evaporate. If a pink color develops before dryness is reached, cobalt is indicated. Continue the electrolysis for 30 min. and repeat the test. When no pink color develops upon evaporating the test portion to dryness, the electrolysis is finished. Remove the electrode, wash thoroughly with a stream of water, then dip into fresh alcohol, dry, and weigh. The increase in weight is due to cobalt, nickel, sulfur, and zinc if present (Note).

NOTE.—Nickel may be disregarded unless it is known to be present in appreciable amounts. The extremely small amount present in the cobalt used for preparing the drier may be disregarded. Sulfur is always deposited whenever sulfite is added to the electrolyte, but it need not be determined because it has been experimentally found that when using the current specified above, 0.0030 g. of sulfur is deposited for every gram of metal plated out. This amount is independent of reasonable sulfite concentration, but does increase as the current density increases. If the sulfite is not added, approximately a corresponding amount of oxygen (presumably) is deposited and complete deposition of cobalt is considerably prolonged. Since some driers contain zinc, it is good practice, unless zinc is known to be absent, to test the cobalt deposit as follows: Dissolve the deposit with diluted HNO_3 (1:1), add 5 ml. of diluted H_2SO_4 (1:1), and evaporate to fumes. Add an equal volume of water and again evaporate to fumes. Repeat to insure complete removal of HNO_3. Add 50 ml. of water and warm to dissolve the anhydrous cobalt sulfate. Cool to at least room temperature. Add methyl red indicator, then NH_4OH (1:3) drop by drop until the indicator just turns yellow. Back-titrate with 1 *N* H_2SO_4 to the red color, then add 1 ml. in excess. Pass H_2S into the solution, and gradually dilute to 100 ml. with water. A white precipitate indicates zinc. If present, add about 5 ml. of gelatin solution (0.02 per cent), stir well, and let stand at least 1 hr. Filter, and wash with cool water. Ash at a temperature below 800 C. to constant weight. The zinc oxide thus obtained may be contaminated with cobalt. The amount of contamination can then be determined colorimetrically by dissolving the zinc oxide with a measured amount of HCl (sp. gr. 1.19) and comparing the blue color with that obtained by adding known amounts of cobalt to the same volume of HCl.

[4] A type of apparatus which has been found suitable is that shown in the catalog of the Will Corporation, and known as the "Braun Series Circuit Electrolytic Apparatus," using platinum anodes and cathodes. The platinum electrodes are available from any of the usual sources and are of designated areas. A rotating cathode can be used, but its only advantage is one of speed. A suitable apparatus can be set up using a storage battery, a rheostat, an ammeter, and a binding post to hold the electrodes.

Zinc

Reagent

16. *Standard Potassium Ferrocyanide Solution.*—Dissolve 40 g. of $K_4Fe(CN)_6$ in distilled water and dilute to 1 liter. Allow the solution to stand for about a month before using. Standardize against pure zinc in the same manner as directed for the determination of zinc in liquid driers (Section 17), using approximately 0.2 g. of zinc for a titration.

Procedure

17. (*a*) Weigh accurately 10 to 15 g. of the sample into a 250-ml. Erlenmeyer flask. Dilute to a volume of 50 to 70 ml. with petroleum spirits and shake vigorously with 15 ml. of HCl (sp. gr. 1.19) and 30 ml. of distilled water. Pour the contents of the flask into a 500-ml. separatory funnel and drain the lower aqueous layer into a beaker. Repeat the above extractions twice, using half the amount of acid and water. Finally wash with 30 ml. of water, collecting the lower layers in a beaker. Dilute the combined aqueous layers to 500 ml. in a volumetric flask.

(*b*) Pipette accurately 50 ml. of the acid solution into a beaker, dilute with 50 ml. of water, make slightly alkaline with NH_4OH, and then add exactly 3 ml. of HCl (sp. gr. 1.19). Heat to about 80 C. and titrate with standard $K_4Fe(CN)_6$, using uranium nitrate solution (10 per cent) as an external indicator. The desired end point is a brown tinge obtained by adding a drop of the titrated solution to a drop of the indicator on a white glazed tile.

Iron

Reagents

18. (*a*) *Stannous Chloride Solution.*—Dissolve 50 g. of $SnCl_2 \cdot 2H_2O$ in 100 ml. of HCl (sp. gr. 1.19) and dilute to 1000 ml. Keep a few pieces of metallic tin in the bottle.

(*b*) *Reinhardt Solution.*—Dissolve 200 g. of $MnSO_4 \cdot 4H_2O$ in 1 liter of water and add to a cooled mixture of the following: 400 ml. of H_2SO_4, (sp. gr. 1.84), 1200 ml. of water, and 400 ml. of H_3PO_4 (85 per cent).

Procedure

19. Weigh 10 g. of the sample into a 200-ml. tall-form beaker, and add 100 ml. of HCl (1:4). Heat the mixture to just below the boiling point for 1 hr., occasionally stirring vigorously during this time. By this time most, if not all, of the iron has been extracted. Add 5 g. of beeswax, stir well until the wax melts, and cool somewhat below the solidification point of the wax. Break up the solidified wax and filter through paper, catching the filtrate in a 250-ml. volumetric flask. Wash moderately with cool water. Dry the wax and paper, then carefully ash in a porcelain crucible. Dissolve the ash by warming with HCl (sp. gr. 1.19) and wash into the main portion. Dilute the solution to 250 ml. and transfer a 50-ml. aliquot to a 150-ml. beaker. Concentrate the 50-ml. portion to about 25 ml. and, while still hot, add stannous chloride solution drop by drop until colorless, then one drop in excess. Cool to room temperature and add at once 10 ml. of saturated $HgCl_2$ solution. Let stand 15 min., stirring occasionally during this time. Wash into a large casserole. Add 25 ml. of Reinhardt solution and dilute with cool water to about 500 ml. Titrate with 0.1 *N* $KMnO_4$ to the same shade of pink as obtained in a blank test made on the reagent. One milliliter of 0.1 *N* $KMnO_4$ is equivalent to 0.005584 g. of iron.

Standard Method of Test for

ACID NUMBER OF ROSIN[1]

A.S.T.M. Designation: D 465 – 42

ADOPTED, 1942.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 465; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test is intended for determining the acid number of rosin.

NOTE.—Due to difficulty in observing the end point in the titration, because of the dark color of the solution, this method using an internal indicator is not recommended for use with rosins of grades darker than G, that is, of grades D, E, F, and FF.

Special Solutions Required

2. (*a*) *Sodium Hydroxide Solution (0.5 N).*—Accurately standardize a 0.5 *N* NaOH solution.

(*b*) *Ethyl Alcohol.*—Ninety-five per cent neutral ethyl alcohol, or neutral specially denatured alcohol (formula No. 30).

(*c*) *Phenolphthalein Solution.*—Dissolve 1 g. of phenolphthalein in 100 ml. of 95 per cent specially denatured alcohol, and neutralize the slightly acid alcohol solution with a caustic soda solution.

Procedure

3. In a 250-ml. Erlenmeyer flask[3] of heat-resistant glass,[4] dissolve 1.95 to 2.05 ± 0.001 g. of the sample[5] of rosin in 50 ml. of neutral alcohol. Heat to dissolve the rosin and titrate the hot solution with standard 0.5 *N* NaOH solution using 1 ml. of phenolphthalein solution as indicator. The standard 0.5 *N* NaOH solution should be added as rapidly as possible, preferably with a wide-open burette until the end point is approached, and then by rapid dropping until the end point is reached.[6]

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-17 on Naval Stores.

[2] Prior to adoption as standard, this method was published as tentative from 1937 to 1942, being revised in 1942.

[3] The flasks should be either new or cleaned by rinsing with a hot solution of hydrofluoric acid (2 to 3 per cent). This removes from the flasks the adhering partially disintegrated silicates which would interfere with the determination. This treatment of flasks is not as important for determination of acid number as for determination of saponification number; however, it should be a good precautionary measure.

[4] Pyrex glass is very satisfactory for this purpose.

[5] It is desirable to stay within rather narrow limits as to size of sample, in order not to introduce too large variations in the relative concentrations of rosin, alcohol, and alkali solutions.

[6] Rapid titration is desirable to prevent any saponification of neutral constituents which slow titration might entail.

Calculation

4. The acid number, expressed in milligrams of KOH per gram, shall be calculated from the following formula:

$$\text{Acid number} = \frac{\text{ml. of NaOH} \times N \times 56.1}{\text{wt. of sample in grams}}$$

where N = the normality of the NaOH solution used.

Standard Method of Test for

SAPONIFICATION NUMBER OF ROSIN[1]

A.S.T.M. Designation: D 464 - 42

ADOPTED, 1942.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 464; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test is intended for determining the saponification number of rosin.

NOTE.—Due to difficulty in observing the end point in the titration, because of the dark color of the solution, this method using an internal indicator is not recommended for use with rosins of grades darker than G, that is, of grades D, E, F, and FF.

Special Solutions Required

2. (*a*) *Hydrochloric Acid Solution (0.5 N).*—Accurately standardize a 0.5 *N* HCl solution.

(*b*) *Alcoholic Potassium Hydroxide Solution.*—Dissolve 40 g. of c. p. KOH in 1 liter of 95 per cent redistilled specially denatured alcohol (formula No. 30). The alcohol shall be redistilled from KOH over which it has been standing for some time, or with which it has been boiled for some time, under a reflux condenser. The solution must be clear. If it contains insoluble matter, it shall be either decanted after settling overnight or filtered.

(*c*) *Phenolphthalein Solution.*—Dissolve 1 g. of phenolphthalein in 100 ml. of 95 per cent specially denatured alcohol (formula No. 30), and neutralize the slightly acid alcohol solution with NaOH solution.

Procedure

3. (*a*) Weigh 2.45 to 2.55 ± 0.001 g. of the sample[3] of rosin into a 250-ml. Erlenmeyer flask[4] of heat-resistant glass.[5] Dissolve the sample in 25 ml. of alcohol (formula No. 30) and into it pipette[6] 25 ml. of the alcoholic KOH solution, allowing the pipette to drain for a definite time. Connect the flask preferably to a water condenser or to an air condenser made from glass tubing 5 mm. in inside diameter, having a minimum length of

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-17 on Naval Stores.

[2] Prior to adoption as standard, this method was published as tentative from 1937 to 1942, being revised in 1942.

[3] It is desirable to stay within rather narrow limits as to size of sample, in order not to introduce too large variations in the relative concentrations of rosin, alcohol, and alkali solutions.

[4] The flasks should be either new or cleaned by rinsing with a hot solution of hydrofluoric acid (2 to 3 per cent). This removes from the flasks the adhering partially disintegrated silicates which would interfere with the determination.

[5] Pyrex glass is very satisfactory for this purpose.

[6] The use of a Lowy automatic pipette is recommended.

32 in. Place on a *hot* hot-plate and boil the solution gently for exactly 1 hr., taking the starting time at the point of incipient boiling. Cool and without washing down the condenser titrate immediately with 0.5 *N* hydrochloric acid solution using 1 ml. of phenolphthalein indicator.

(*b*) Conduct two or three blank determinations on 25-ml. portions of alcohol (formula No. 30) with 25-ml. portions of the alcoholic KOH solution, using the same pipette and draining for the same length of time as above.

Calculation

4. The saponification number shall be calculated from the following formula:

$$\text{Saponification number} = \frac{(B - S) \times N \times 56.1}{W}$$

where:

B = milliliters of HCl for titration of blank,

S = milliliters of HCl for titration of sample,

N = normality of standard HCl solution used in titration, and

W = weight of sample in grams.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: K21.1-1936

Standard Method of Test for

TOLUENE INSOLUBLE SOLID MATTER IN ROSIN[1]

CHIEFLY SAND, CHIPS, DIRT, AND BARK

A.S.T.M. Designation: D 269 - 30

ADOPTED, 1930.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 269; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test covers a procedure for determining the amount of solid matter (chiefly sand, chips, dirt, and bark) in rosin insoluble in toluene.

Treatment of Sample

2. (*a*) If the sample is less than 200 g., powder it, immediately before the determination is made, to pass a No. 10 (2000-micron) sieve,[3] mix thoroughly, and place in a wide-mouth bottle which is completely filled thereby.

(*b*) If the sample is more than 200 g., crush it, immediately before the determination is made, to pass a $\frac{1}{2}$-in. sieve,[3] mix, divide by quartering to about 200 g., and treat as described in Paragraph (*a*).

Procedure

3. Place 50 g. of the freshly-powdered sample in a 300-ml. beaker, add 150 ml. of toluene, free from water and non-volatile residue, and dissolve the sample with the aid of heat and occasional shaking. When solution is apparently complete (no particles of rosin visible), filter at once through a 25-ml. porcelain Gooch crucible which has been previously prepared with a mat of pure well-washed asbestos (such as is used for the determination of barium sulfate), and which has been finally washed thoroughly with toluene, dry at 105 to 110 C. for 30 min., cool in a desiccator, and weigh. If the rosin filtrate is not clear, return it through the Gooch crucible until it is clear, finally washing the residue and the outside of the crucible free from rosin with additional hot toluene. Dry the crucible and contents to constant weight at 105 to 110 C. (1 hr. usually suffices), cool in a desiccator, weigh, and calculate the percentage of solid matter insoluble in toluene.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-17 on Naval Stores.

[2] Prior to adoption as standard, this method was published as tentative from 1927 to 1930. Editorially revised and rearranged in 1939.

[3] Detailed requirements for this sieve are given in the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for

ACETONE[1]

A.S.T.M. Designation: D 329 - 33

ADOPTED, 1933.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 329; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover acetone.

Properties

2. Acetone shall conform to the following requirements:

Purity	not less than 98 per cent dimethylketone
Specific gravity, 20/20 C.	0.791 to 0.799
Color	water white
Distillation range:	
Below 55.0 C.	none
Above 57.5 C.	none
Nonvolatile matter	not more than 0.005 g. per 100 ml.
Water	miscible without turbidity with 19 volumes of 60° A.P.I. gasoline at 20 C.
Acidity (free acid as CO_2)	not more than 0.002 per cent by weight, equivalent to 0.051 mg. of KOH per gram of sample
Water solubility	miscible with distilled water in all proportions
Alkalinity	not alkaline to para-nitrophenol
Potassium permanganate test	color of added $KMnO_4$ to be retained at least 30 min.

Methods of Testing

3. The material shall be sampled and the properties enumerated in these specifications determined in accordance with the Standard Methods of Sampling and Testing Lacquer Solvents and Diluents (A.S.T M. Designation: D 268) of the American Society for Testing Materials.[3]

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

[2] Prior to adoption as standard, these specifications were published as tentative from 1931 to 1933. Editorially revised and rearranged in 1939.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for
AMYL ALCOHOL (SYNTHETIC)[1]

A.S.T.M. Designation: D 319 – 40

ADOPTED, 1933; REVISED, 1940.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 319; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover amyl alcohol (synthetic).

Properties

2. Amyl alcohol (synthetic) shall conform to the following requirements:

Specific gravity, 20/20 C	0.812 to 0.820
Color	water white
Distillation range:	
Below 118 C	none
Below 120 C	not more than 5 per cent
Below 125 C	not more than 50 per cent
Below 130 C	not more than 85 per cent
Above 140 C	none
Nonvolatile matter	not more than 0.005 g. per 100 ml.
Odor	mild, nonresidual
Water	miscible without turbidity with 19 volumes of 60° A.P.I. gasoline at 20 C.
Acidity	none

Methods of Testing

3. The material shall be sampled and the properties enumerated in these specifications determined in accordance with the Standard Methods of Sampling and Testing Lacquer Solvents and Diluents (A.S.T.M. Designation: D 268) of the American Society for Testing Materials.[3]

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

[2] Prior to adoption as standard, these specifications were published as tentative from 1930 to 1933, being revised in 1930. Editorially revised in 1942.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for

AMYL ACETATE (SYNTHETIC) (85 to 88 PER CENT GRADE)[1]

A.S.T.M. Designation: D 318 – 39

ADOPTED, 1933; REVISED, 1936, 1939.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 318; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover amyl acetate (synthetic) (85 to 88 per cent grade).

Properties

2. Amyl acetate (synthetic) shall conform to the following requirements:

Specific gravity, 20/20 C	0.860 to 0.870
Color	water white
Distillation range:	
Below 126 C	none
Below 130 C	not more than 5 per cent
Below 135 C	not more than 25 per cent
Below 140 C	not more than 75 per cent
Above 155 C	none
Nonvolatile matter	not more than 0.005 g. per 100 ml.
Odor	mild, nonresidual
Water	miscible without turbidity in all proportions with 60° A.P.I. gasoline at 20 C.
Acidity (free acid as acetic acid)	not more than 0.03 per cent by weight, equivalent to 0.28 mg. of KOH per gram of sample
Ester value	85 to 88 per cent by weight

Methods of Testing

3. The material shall be sampled and the properties enumerated in these specifications determined in accordance with the Standard Methods of Sampling and Testing Lacquer Solvents and Diluents (A.S.T.M. Designation: D 268) of the American Society for Testing Materials.[3]

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

[2] Prior to adoption as standard, these specifications were published as tentative from 1930 to 1933.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for

AMYL ACETATE MADE FROM FUSEL OIL (85 to 88 PER CENT GRADE)[1]

A.S.T.M. Designation: D 554 - 39

ADOPTED, 1939.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 554; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover amyl acetate made from fusel oil (85 to 88 per cent grade).

Properties

2. Amyl acetate (made from fusel oil) shall conform to the following requirements:

Specific gravity, 20/20 C	0.860 to 0.865
Color	water white
Distillation range:	
Below 110 C	none
Below 120 C	not more than 15 per cent
Below 130 C	not more than 50 per cent
Below 140 C	not less than 60 per cent
Above 150 C	none
Nonvolatile matter	not more than 0.005 g. per 100 ml.
Odor	mild, nonresidual
Water	miscible without turbidity with 19 volumes of 60° A.P.I. gasoline at 20 C.
Acidity (free acid as acetic acid)	not more than 0.03 per cent by weight, equivalent to 0.28 mg. of KOH per gram of sample
Ester value	85 to 88 per cent by weight

Methods of Testing

3. The material shall be sampled and the properties enumerated in these specifications determined in accordance with the Standard Methods of Sampling and Testing Lacquer Solvents and Diluents (A.S.T.M. Designation: D 268) of the American Society for Testing Materials.[3]

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

[2] These specifications were revised and issued under the designation D 554 in 1939. They formerly constituted a portion of the Standard Specifications for Normal Amyl Acetate (D 318 - 36) which was published as standard in 1933, and revised in 1936.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for

INDUSTRIAL 90 PER CENT BENZENE (BENZOL)[1]

A.S.T.M. Designation: D 361 – 36

ADOPTED, 1936.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 361; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover industrial 90 per cent benzene (benzol).

Properties

2. Industrial 90 per cent benzene (benzol) shall conform to the following requirements:

Specific gravity, 20/20 C	0.868 to 0.882
Color	water white
Distillation range:	
Below 77 C	none
Below 100 C	not less than 92 per cent
Above 122 C	none
Nonvolatile matter	not more than 0.005 g. per 100 ml.
Odor	characteristic aromatic hydrocarbon, nonresidual
Water	not sufficient to show turbidity at 20 C.
Acidity	none
Sulfur as H_2S and SO_2	none
Corrosion:	
Copper corrosion test	copper shall show no iridescence
Mercury test	mercury shall show no discoloration

Methods of Testing

3. The material shall be sampled and the properties enumerated in these specifications determined in accordance with the Standard Methods of Sampling and Testing Lacquer Solvents and Diluents (A.S.T.M. Designation: D 268) of the American Society for Testing Materials.[3]

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1933 to 1936. Editorially revised and rearranged in 1939.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for
BUTANOL (NORMAL BUTYL ALCOHOL)[1]

A.S.T.M. Designation: D 304 – 40

ADOPTED, 1933; REVISED, 1940.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 304; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover butanol (normal butyl alcohol).

Properties

2. Butanol (normal butyl alcohol) shall conform to the following requirements:

Specific gravity, 20/20 C	0.810 to 0.815
Color	water white
Distillation range:	
Below 100 C	none
Below 105 C	not more than 2 per cent
Below 115 C	not more than 10 per cent
Above 118 C	none
Nonvolatile matter	not more than 0.005 g. per 100 ml.
Odor	mild, nonresidual
Water	miscible without turbidity with 19 volumes of 60° A.P.I. gasoline at 20 C.
Acidity (free acid as acetic acid)	not more than 0.03 per cent by weight, equivalent to 0.28 mg. of KOH per gram of sample

Methods of Testing

3. The material shall be sampled and the properties enumerated in these specifications determined in accordance with the Standard Methods of Sampling and Testing Lacquer Solvents and Diluents (A.S.T.M. Designation: D 268) of the American Society for Testing Materials.[3]

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

[2] Prior to adoption as standard, these specifications were published as tentative from 1929 to 1933, being revised in 1930.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for

NORMAL BUTYL ACETATE (88 TO 92 PER CENT GRADE)[1]

A.S.T.M. Designation: D 303 – 40

ADOPTED, 1933; REVISED, 1940.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 303; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover normal butyl acetate (88 to 92 per cent grade).

Properties

2. Normal butyl acetate (88 to 92 per cent grade) shall conform to the following requirements:

Specific gravity, 20/20 C	0.872 to 0.878
Color	water white
Distillation range:	
Below 110 C	none
Below 120 C	not more than 15 per cent
Above 127 C	not more than 30 per cent
Above 145 C	none
Nonvolatile matter	not more than 0.005 g. per 100 ml.
Odor	mild, nonresidual
Water	miscible without turbidity with 19 volumes of 60° A.P.I. gasoline at 20 C.
Acidity (free acid as acetic acid)	not more than 0.03 per cent by weight, equivalent to 0.28 mg. of KOH per gram of sample
Ester value	88 to 92 per cent by weight

Methods of Testing

3. The material shall be sampled and the properties enumerated in these specifications determined in accordance with the Standard Methods of Sampling and Testing Lacquer Solvents and Diluents (A.S.T.M. Designation: D 268) of the American Society for Testing Materials.[3]

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

[2] Prior to adoption as standard, these specifications were published as tentative from 1929 to 1933, being revised in 1930.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for

BUTYL PROPIONATE (90 TO 93 PER CENT GRADE)[1]

A.S.T.M. Designation: D 320 – 40

ADOPTED, 1933; REVISED, 1940.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 320; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover butyl propionate (90 to 93 per cent grade).

Properties

2. Butyl propionate (90 to 93 per cent grade) shall conform to the following requirements:

Specific gravity, 20/20 C	0.872 to 0.878
Color	water white
Distillation range:	
Below 115 C	none
Below 130 C	not more than 20 per cent
Below 138 C	not more than 60 per cent
Above 168 C	none
Nonvolatile matter	not more than 0.005 g. per 100 ml.
Odor	mild, nonresidual
Water	miscible without turbidity with 19 volumes of 60° A.P.I. gasoline at 20 C.
Acidity (free acid as propionic acid)	not more than 0.02 per cent by weight, equivalent to 0.15 mg. of KOH per gram of sample
Ester value	90 to 93 per cent by weight

Methods of Testing

3. The material shall be sampled and the properties enumerated in these specifications determined in accordance with the Standard Methods of Sampling and Testing Lacquer Solvents and Diluents (A.S.T.M. Designation: D 268) of the American Society for Testing Materials.[3]

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

[2] Prior to adoption as standard, these specifications were published as tentative from 1930 to 1933.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for

DIBUTYLPHTHALATE[1]

A.S.T.M. Designation: D 608 - 43

ADOPTED, 1943.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 608; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover dibutylphthalate.

Properties

2. Dibutylphthalate shall conform to the following requirements:

Specific gravity, 20/20 C	1.047 to 1.049
Appearance	clear and free of suspended matter
Color	water white
Odor	substantially none
Water	miscible without turbidity with 19 volumes of 60° A.P.I. gasoline at 20 C.
Acidity (free acid as phthalic acid)	not more than 0.01 per cent by weight, equivalent to 0.067 mg. of KOH per gram of sample
Ester value	not less than 99 per cent by weight, 4-hr. heating period on the steam bath

Methods of Testing

3. The material shall be sampled and the properties enumerated in these specifications determined in accordance with the Standard Methods of Sampling and Testing Lacquer Solvents and Diluents (A.S.T.M. Designation: D 268) of the American Society for Testing Materials.[3]

[1] Under the standardization procedure of the Society these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

[2] Prior to adoption as standard, these specifications were published as tentative from 1941 to 1943.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for
ETHYL ACETATE (85 TO 88 PER CENT GRADE)[1]

A.S.T.M. Designation: D 302 – 33

ADOPTED, 1933.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 302; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover ethyl acetate (85 to 88 per cent grade).

Properties

2. Ethyl acetate (85 to 88 per cent grade) shall conform to the following requirements:

Specific gravity, 20/20 C	0.883 to 0.888
Color	water white
Distillation range:	
Below 70 C	none
Below 72 C	not more than 10 per cent
Above 80 C	none
Nonvolatile matter	not more than 0.005 g. per 100 ml.
Odor	mild, nonresidual
Water	miscible without turbidity with 10 volumes of 60° A.P.I. gasoline at 20 C.
Acidity (free acid as acetic acid)	not more than 0.02 per cent by weight, equivalent to 0.19 mg. of KOH per gram of sample
Ester value	85 to 88 per cent by weight

Methods of Testing

3. The material shall be sampled and the properties enumerated in these specifications determined in accordance with the Standard Methods of Sampling and Testing Lacquer Solvents and Diluents (A.S.T.M. Designation: D 268) of the American Society for Testing Materials.[3]

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

[2] Prior to adoption as standard, these specifications were published as tentative from 1929 to 1933, being revised in 1930. Editorially revised and rearranged in 1939.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for
ETHYL LACTATE (SYNTHETIC)[1]

A.S.T.M. Designation: D 321 – 40

ADOPTED, 1933; REVISED, 1940.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 321; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover ethyl lactate (synthetic).

Properties

2. Ethyl lactate (synthetic) shall conform to the following requirements:

Specific gravity, 20/20 C	1.020 to 1.036
Color	water white
Distillation range:	
Below 102 C	none
Below 130 C	not more than 10 per cent
Below 155 C	not less than 90 per cent
Above 173 C	none
Nonvolatile matter	not more than 0.005 g. per 100 ml.
Odor	mild, nonresidual
Water	miscible without turbidity with 19 volumes of 60° A.P.I. gasoline at 20 C.
Acidity (free acid as lactic acid)	not more than 0.08 per cent by weight, equivalent to 0.5 mg. of KOH per gram of sample
Ester value	96 to 100 per cent by weight

Methods of Testing

3. The material shall be sampled and the properties enumerated in these specifications determined in accordance with the Standard Methods of Sampling and Testing Lacquer Solvents and Diluents (A.S.T.M. Designation: D 268) of the American Society for Testing Materials.[3]

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

[2] Prior to adoption as standard, these specifications were published as tentative from 1930 to 1933

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for
ETHYLENE GLYCOL MONOETHYL ETHER[1,2]

A.S.T.M. Designation: D 331 - 35

ADOPTED, 1935.[3]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 331; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover ethylene glycol monoethyl ether.

Properties

2. Ethylene glycol monoethyl ether shall conform to the following requirements:

Specific gravity, 20/20 C	0.927 to 0.933
Color	water white
Distillation range:	
Below 128 C	none
Below 132 C	not more than 5 per cent
Below 136 C	not less than 95 per cent
Above 137 C	none
Nonvolatile matter	not more than 0.005 g. per 100 ml.
Odor	mild, nonresidual
Water	miscible without turbidity with 19 volumes of 60° A.P.I. gasoline at 20 C.
Acidity	not more than 0.2 mg. of KOH per gram of sample

Methods of Testing

3. The material shall be sampled and the properties enumerated in these specifications determined in accordance with the Standard Methods of Sampling and Testing Lacquer Solvents and Diluents (A.S.T.M. Designation: D 268) of the American Society for Testing Materials.[4]

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

[2] This compound is also known under the names beta-ethoxy ethanol and ethyl glycol.

[3] Prior to adoption as standard, these specifications were published as tentative from 1932 to 1935. Editorially revised in 1939 and 1942.

[4] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for

ACETATE ESTER OF ETHYLENE GLYCOL MONOETHYL ETHER (95 TO 96 PER CENT GRADE)[1,2]

A.S.T.M. Designation: D 343 - 35

ADOPTED, 1935.[3]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 343; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover the acetate ester of ethylene glycol monoethyl ether (95 to 96 per cent grade).

Properties

2. The acetate of ethylene glycol monoethyl ether (95 to 96 per cent grade) shall conform to the following requirements:

Specific gravity, 20/20 C	0.968 to 0.976
Color	not darker than a solution of 0.008 g. of $K_2Cr_2O_7$ per liter of water
Distillation range:	
Below 145 C	none
Between 150 and 160 C	not less than 90 per cent
Above 165 C	none
Nonvolatile matter	not more than 0.03 g. per 100 ml.
Odor	mild, nonresidual
Water	miscible without turbidity with 19 volumes of 60° A.P.I. gasoline at 20 C.
Acidity (free acid as acetic acid)	not more than 0.024 per cent by weight, equivalent to 0.2 mg. of KOH per gram of sample
Ester value	95 to 96 per cent by weight

Methods of Testing

3. The material shall be sampled and the properties enumerated in these specifications determined in accordance with the Standard Methods of Sampling and Testing Lacquer Solvents and Diluents (A.S.T.M. Designation: D 268) of the American Society for Testing Materials.[4]

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

[2] This compound is also known under the names beta-ethoxy ethyl acetate and ethyl glycol acetate.

[3] Prior to adoption as standard, these specifications were published as tentative from 1932 to 1935. Editorially revised and rearranged in 1939.

[4] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for
ETHYLENE GLYCOL MONOBUTYL ETHER[1,2]

A.S.T.M. Designation: D 330 – 35

ADOPTED, 1935.[3]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 330; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover ethylene glycol monobutyl ether.

Properties

2. Ethylene glycol monobutyl ether shall conform to the following requirements:

Specific gravity, 20/20 C	0.899 to 0.905
Color	water white
Distillation range:	
Below 163 C	none
Below 167 C	not more than 5 per cent
Below 171 C	not less than 95 per cent
Above 174 C	none
Nonvolatile matter	not more than 0.005 g. per 100 ml.
Odor	mild, nonresidual
Water	miscible without turbidity with 19 volumes of 60° A.P.I. gasoline at 20 C.
Acidity	not more than 0.2 mg. of KOH per gram of sample

Methods of Testing

3. The material shall be sampled and the properties enumerated in these specifications determined in accordance with the Standard Methods of Sampling and Testing Lacquer Solvents and Diluents (A.S.T.M. Designation: D 268) of the American Society for Testing Materials.[4]

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

[2] This compound is also known under the names beta-butoxy ethanol and butyl glycol.

[3] Prior to adoption as standard, these specifications were published as tentative from 1932 to 1935. Editorially revised in 1939 and 1942.

[4] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for

ISOPROPYL ACETATE[1]

A.S.T.M. Designation: D 657 – 44

ADOPTED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 657; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover two grades of isopropyl acetate, as follows: 85 to 88 per cent grade, and 95 to 98 per cent grade.

Properties

2. Isopropyl acetate shall conform to the requirements prescribed in Table I.

Methods of Testing

3. The material shall be sampled and the properties enumerated in these specifications determined in accordance with the Standard Methods of Sampling and Testing Lacquer Solvents and Diluents (A.S.T.M. Designation: D 268) of the American Society for Testing Materials.[3]

TABLE I.—REQUIREMENTS FOR ISOPROPYL ACETATE.

	85 to 88 per cent Grade	95 to 98 per cent Grade
Specific gravity, 20/20 C.	0.855 to 0.862	0.866 to 0.873
Color	water white	water white
Distillation range:		
None below	81 C.	85 C.
None above	93 C.	90 C.
Nonvolatile matter	not more than 0.005 g. per 100 ml.	not more than 0.005 g. per 100 ml.
Odor	mild, nonresidual	mild, nonresidual
Water	miscible without turbidity with 19 volumes of 60° A.P.I. gasoline at 20 C.	miscible without turbidity with 19 volumes of 60° A.P.I. gasoline at 20 C.
Acidity (free acid as acetic acid)	not more than 0.02 per cent by weight, equivalent to 0.19 mg. of KOH per gram of sample	not more than 0.02 per cent by weight, equivalent to 0.19 mg. of KOH per gram of sample
Ester value	not less than 85 per cent by weight	not less than 95 per cent by weight

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1942 to 1944.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for

INDUSTRIAL PURE TOLUENE (TOLUOL)[1]

A.S.T.M. Designation: D 362 – 36

ADOPTED, 1936.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 362; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover industrial pure toluene (toluol).

Properties

2. Industrial pure toluene (toluol) shall conform to the following requirements:

Specific gravity, 20/20 C	0.860 to 0.870
Color	water white
Distillation range:	
Below 107.5 C	none
Above 112.5 C	none
The total distillation range of any specific sample shall not be greater than 3.0 C.	
Nonvolatile matter	not more than 0.005 g. per 100 ml.
Odor	characteristic aromatic hydrocarbon, nonresidual
Water	not sufficient to show turbidity at 20 C.
Acidity	none
Sulfur as H_2S and SO_2	none
Corrosion:	
Copper corrosion test	copper shall show no iridescence
Mercury test	mercury shall show no discoloration

Methods of Testing

3. The material shall be sampled and the properties enumerated in these specifications determined in accordance with the Standard Methods of Sampling and Testing Lacquer Solvents and Diluents (A.S.T.M. Designation: D 268) of the American Society for Testing Materials.[3]

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1933 to 1936. Editorially revised and rearranged in 1939.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for

TRICRESYL PHOSPHATE[1, 2]

A.S.T.M. Designation: D 363 – 36

ADOPTED, 1936.[3]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 363; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover tricresyl phosphate.

Properties

2. Tricresyl phosphate shall conform to the following requirements:

Specific gravity, 20/20 C	1.160 to 1.180
Color	not more than 100 on the Hazen platinum-cobalt standard
Volatile matter	not more than 0.20 per cent
Free phenols	not more than 0.05 per cent
Potassium permanganate test	no change in color after standing 30 min.
Ester value[a]	not less than 99 per cent by weight
Odor	substantially none
Water	miscible without turbidity with 19 volumes of 60° A.P.I. gasoline at 20 C.
Acidity	not more than 0.1 mg. of KOH per gram of sample

[a] Ester value shall be determined in accordance with Section 11 of Standard Methods D 268[4] except that the heating period shall be from 8 to 16 hr.

Methods of Testing

3. The material shall be sampled and the properties enumerated in these specifications determined in accordance with the Standard Methods of Sampling and Testing Lacquer Solvents and Diluents (A.S.T.M. Designation: D 268) of the American Society for Testing Materials.[4]

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

[2] Prepared from commercial cresylic acid.

[3] Prior to adoption as standard, these specifications were published as tentative from 1933 to 1936. Editorially revised in 1939 and 1942.

[4] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications for

INDUSTRIAL XYLENE (XYLOL) OR SOLVENT NAPHTHA[1]

A.S.T.M. Designation: D 364 - 36

ADOPTED, 1936.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 364; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover industrial xylene (xylol) or solvent naphtha.

Properties

2. Industrial xylene (xylol) or solvent naphtha shall conform to the following requirements:

Specific gravity, 20/20 C	0.856 to 0.867
Color	water white
Distillation range:	
Below 123 C	none
Below 129 C	not more than 5 per cent
Below 143 C	not less than 90 per cent
Above 160 C	none
Nonvolatile matter	not more than 0.005 g. per 100 ml.
Odor	characteristic aromatic hydrocarbon, nonresidual
Water	not sufficient to show turbidity at 20 C.
Acidity	none
Sulfur as H_2S and SO_2	none
Corrosion:	
Copper corrosion test	copper shall show no iridescence
Mercury test	mercury shall show no discoloration

Methods of Testing

3. The material shall be sampled and the properties enumerated in these specifications determined in accordance with the Standard Methods of Sampling and Testing Lacquer Solvents and Diluents (A.S.T.M. Designation: D 268) of the American Society for Testing Materials.[3]

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1933 to 1936. Editorially revised and rearranged in 1939.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Specifications and Tests for

SOLUBLE NITROCELLULOSE[1]

A.S.T.M. Designation: D 301 - 33

ADOPTED, 1933.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 301; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover the material known as soluble nitrocellulose (also known as soluble cotton), which is shipped wet to conform to the regulations of the Interstate Commerce Commission.

Properties

2. (*a*) *Appearance.*—The nitrocellulose shall not be discolored and shall be free from lumps and foreign matter, such as charred particles.

(*b*) *Ash.*—The ash content shall not exceed 0.30 per cent, calculated on the basis of dry-weight soluble nitrocellulose.

(*c*) *Nitrogen.*—The percentage of nitrogen, calculated on the basis of dry-weight soluble nitrocellulose, shall be within the limits agreed upon by the purchaser and the seller for the particular type of soluble nitrocellulose.

(*d*) *Stability.*—The stability as determined by the 134.5 C. test shall not be less than 25 min.

(*e*) *Consistency* (*Viscosity*).—The consistency shall be within the limits agreed upon by the purchaser and the seller for the particular type of soluble nitrocellulose.

(*f*) *Solubility and Appearance of the Solution.*—The solubility and appearance of the sample shall be equal to the reference standard for the particular type of soluble nitrocellulose.

(*g*) *Film Test.*—The film test of the sample shall be equal to that of the reference standard for the particular type of soluble nitrocellulose.

(*h*) *Toluol Dilution Test.*—The toluol dilution value of the sample shall be equivalent to that of the reference standard for the particular type of soluble nitrocellulose.

SAMPLING AND METHODS OF TESTING

Sampling

3. Samples shall be taken from not less than 10 per cent (at least two

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

[2] Prior to adoption as standard, these specifications were published as tentative from 1929 to 1933, being revised in 1930 and 1931. Editorially revised and rearranged in 1939.

barrels) of each lot or batch in the shipment. In sampling the barrels, two samples of approximately 1 pt. each shall be taken from two well separated points at least 1 ft. beneath the surface of the material in the barrel. These samples shall then be composited to represent each lot or batch in the shipment.

ASH

Procedure

4. Weigh approximately 2 g. of sample in a tared and ignited crucible, and dry at 100 C. for approximately 30 min. Determine the dry weight of the sample. Moisten the residue with 20 to 30 drops of c.p. HNO_3 (sp. gr. 1.42) and heat on a steam bath until the sample is decomposed to a gummy mass. If necessary to complete the decomposition, add several additional drops of HNO_3 at intervals. Then heat the crucible slowly over a bunsen burner or in an electric furnace until all volatile matter is driven off. Finally, ignite at red heat to constant weight, cool, and weigh. Calculate the percentage of ash as follows:

$$\text{Percentage of ash} = \frac{\text{wt. of ash}}{\text{wt. of dry sample}} \times 100$$

NOTE.—Danger from inflammable vapor may be avoided by a preliminary air drying of the sample in the event that the number of samples being tested or the construction of the oven are such as to warrant this precaution.

NITROGEN

Apparatus

5. Use the duPont Nitrometer[3] which is illustrated in Figs. 1 to 4. Figure 2 (*b*) shows the universal measuring tube, although the nitrocellulose measuring tube shown in Fig. 2 (*a*) is preferable for testing nitrocellulose. If desired, both measuring tubes may be included in the assembly, by replacing the three-way glass manifold with a four-way manifold.

[3] Drawings reproduced by permission from the Standard Specifications for Laboratory Apparatus Adopted by the Manufacturing Chemists' Association of the United States, Part I.

Procedure

NOTE.—The cellulose acetate mask described in Section 7 (*d*) must be worn during the generation and measurement of the gas as a precaution in case of an explosion. Nitrogen-free c.p. H_2SO_4 shall be used.

6. (*a*) Accurately calibrate the measuring tube in the usual manner, using mercury as the calibrating liquid.

(*b*) Standardize the apparatus using c.p. KNO_3 recrystallized twice from distilled water and then washed with 95 per cent ethyl alcohol, ground to pass a No. 100 (149-micron) sieve, and dried for 2 to 3 hr. at 135 to 150 C. Fill the compensating, measuring, and reaction tubes and their connections with mercury. Run 20 to 30 ml. of H_2SO_4 (94.5 $\pm$ 0.5 per cent) (Note) into the reaction bulb through the cup at the top and admit about 210 ml. of air. Close the stopcocks and shake the bulb well. This desiccates the air which is then run into the compensating tube until the mercury is about on a level with the 12.50 per cent mark on the measuring tube, the two tubes being held at the same height. Then seal the compensating tube using a small, blowpipe flame. Accurately weigh 0.9000 to 1.0000 g. of the KNO_3 and introduce it into the reaction bulb. Wash the cup with 20 ml. of c.p. H_2SO_4 (94.5 $\pm$ 0.5 per cent) (Note) in three or four portions, each portion being run separately into the bulb. During this operation allow the bottom stopcock of the reaction bulb to remain open. Then with the bottom stopcock still open lower the reservoir bulb to give reduced pressure in the reaction bulb and gently shake the reaction bulb to start the decomposition. After the evolution of NO has become slow (it is extremely important that the bottom stopcock be left open until the major part of the decomposition has occurred; otherwise sudden evolution of gas will burst the bulb, scattering

acid and glass) lower the reservoir bulb until all but 25 ml. of the mercury in the reaction bulb is withdrawn, close the bottom stopcock, and shake the reaction bulb vigorously for 5 min. When the reaction is completed allow the gas to cool for 20 min., then transfer the gas to the measuring tube. By means of the leveling device make careful adjustment of the mercury levels so that the mercury in the measuring tube is at the 13.85 per cent mark (the theoretical percentage of nitrogen in potassium nitrate) if an exactly 1.0000-g. sample was used, or a proportional reading if less was used. Paste a strip of paper on the compensating tube at the level of the mercury and the standardization is completed. It is advisable to make several check determinations, preferably on different days, to insure accurate standardization. Determinations should check within plus or minus 0.01 per cent.

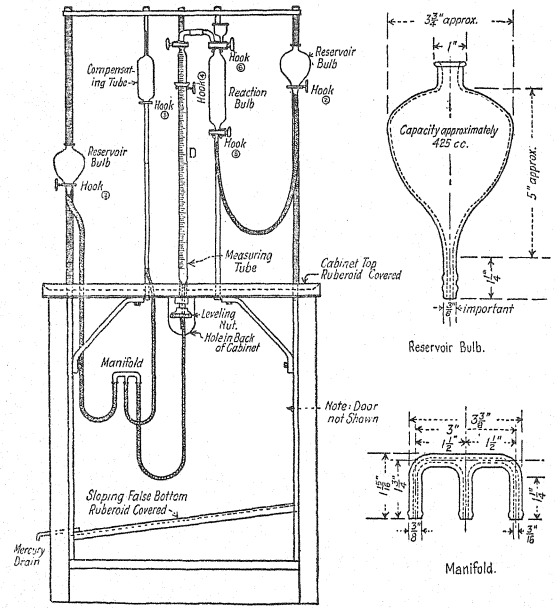
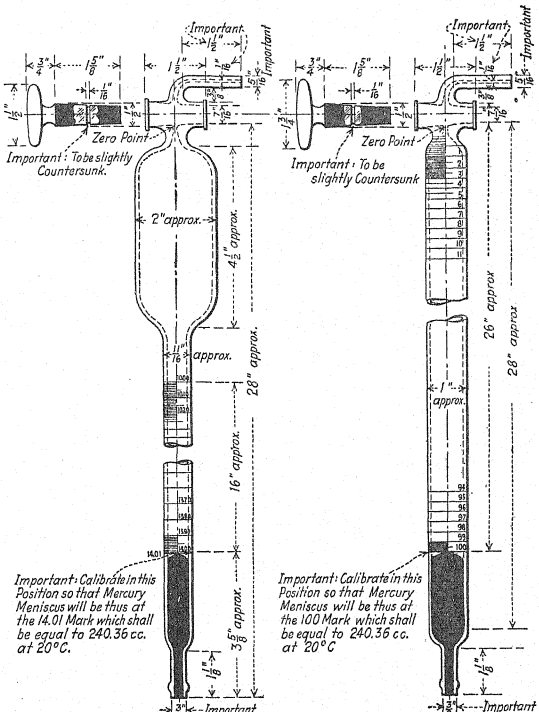


FIG. 1. General Assembly of Apparatus for Nitrogen Determination.

(*c*) Roughly dry the soluble nitrocellulose as for the ash determination described in Section 4 and weigh a sample of from 1.0 to 1.5 g. in a weighing bottle. Dry to constant weight at 98



(*a*) Nitrocellulose tube. (*b*) Universal tube.

FIG. 2.—Measuring Tubes for Nitrogen Determination.

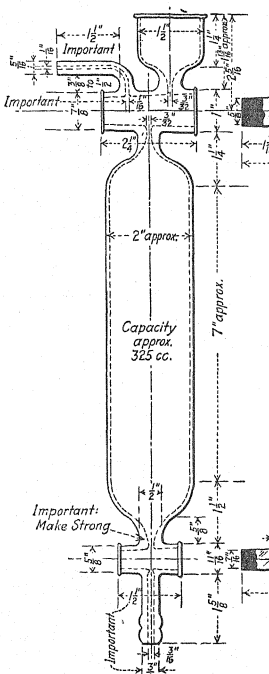


FIG. 3.—Reaction Bulb.

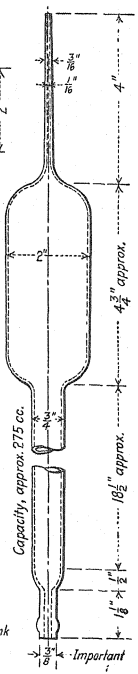


FIG. 4.—Compensating Tube.

to 102 C. (1 to 1½ hr. usually suffices) and cool in a desiccator. Transfer to the cup of the reaction bulb of the nitrometer; then, using c.p. H_2SO_4 (94.5 ± 0.5 per cent) dissolve the sample, withdraw into the reaction bulb, and rinse the cup into the bulb several times. Use a total of 20 ml. of acid for dissolving and rinsing. Complete the determination in accordance with the standardization of the apparatus described in Paragraph (*b*) and take a reading after adjusting the level of the mercury in the reading tube to the mark on the compensating tube. The reading divided by the weight of sample gives the percentage of nitrogen.

STABILITY

Apparatus

7. The apparatus shall consist of the following:

(*a*) *Copper Bath.*—Copper bath with copper or brass condenser, as shown in Figs. 5 and 6. These baths are usually made to hold 13 or 15 test tubes.

(*b*) *Test Tubes.*—Fifteen heat-resistant glass[4] test tubes 290 ± 1 mm. in length with an outside diameter of 18 mm. and a wall thickness of 1.5 mm.

(*c*) *Heater.*—An electric hot plate for heating the bath.

(*d*) *Face Mask.*—A face mask, so constructed that a heavy piece of clear cellulose acetate sheeting protects the face.

(*e*) *Gloves.*—A pair of heavy gloves.

(*f*) *Pincers.*—Long pincers for handling the test tubes.

(*g*) *Thermometer.*—An A.S.T.M. Stability Test Thermometer graduated in Centigrade degrees, having a range of 130 to 140 C., and conforming to the requirements for this thermometer as prescribed in the Standard Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1) of the American Society for Testing Materials.[5]

[4] Pyrex glass is very satisfactory for this purpose.

Special Solution Required

8. *Methyl Violet Test Paper.*—Conduct the preparation and drying of the methyl violet test paper in a room that is free of acid fumes. Prepare a dye solution according to the following formula:

Para-rosaniline[a] (converted to acetate)	0.2500 g.
Crystal violet[a]	0.1680 g.
C.p. glycerol	6 ml.
Distilled water	30 ml.
95 per cent ethyl alcohol	sufficient to make up to 100 ml.

[a] The para-rosaniline acetate used in the foregoing formula is made by adding an excess of glacial acetic acid to 0.2500 g. of para-rosaniline and heating in a porcelain casserole over a steam bath until the excess acid is removed. The other ingredients are then added and the volume made up to a total of 100 ml. with 95 per cent ethyl alcohol. The crystal violet mentioned in the formula may be replaced by Methyl Violet N.E. (E. I. du Pont de Nemours and Co.) or Methyl Violet 2 B (National Aniline and Chemical Co.).

Place a small quantity of the dye solution into a rectangular glass tray that is supported in a tilted position. Slowly draw a quarter sheet of filter paper through the solution and up and over the side of the dish to remove the excess liquid. The filter paper shall be carefully selected and of such properties as to maintain a permanent color when treated. The S. & S. No. 597 filter paper has been found to be the only paper giving consistent results. Holding the sheet horizontally, wave it gently back and forth for about 1 min., then suspend vertically until dry. When dry, cut into strips 70 by 20 mm. and store in a well-stoppered bottle.

Check each batch of prepared test papers against the papers held as standard by testing the same sample of nitrocellulose with both papers.

Procedure

9. (*a*) Conduct the test in a room that is free of acid fumes. Dried nitrocellulose is very inflammable and if ignited by fire, spark, or static electricity will

[5] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

flare up and shatter the test tube. It is important, therefore, that the operator wear the cellulose acetate mask and heavy gloves (Section 7) and that the tubes be handled with long pincers.

(*b*) Maintain the heating bath at a temperature of 134.5 ± 0.5 C. by the use of the required mixture of toluol and xylol. The proper mixture is approximately one part toluol and four parts xylol, but the exact proportions must be determined by the "cut-and-try" method. If the original mixture is too high, add more toluol; if it is too low, allow the condenser to run hot until the desired temperature is reached. On continued operation the temperature will have a tendency to rise, which should be corrected by adding toluol.

(*c*) Weigh accurately 2.5 g. of the sample previously dried at 40 to 45 C. for 4 to 5 hr., or air dried over night followed by a drying at 40 to 45 C. for 30 min. Shake or press the sample down in the test tube so that it occupies the lower 2 in. of the tube and swab out all nitrocellulose particles adhering to the inside wall of the tube. Place the methyl violet test paper in the tube so that its lower edge is 1 in. from the top of the sample in which position the paper must remain throughout the test. Stopper the tube with a cork provided with a hole or notch 4 mm. in diameter

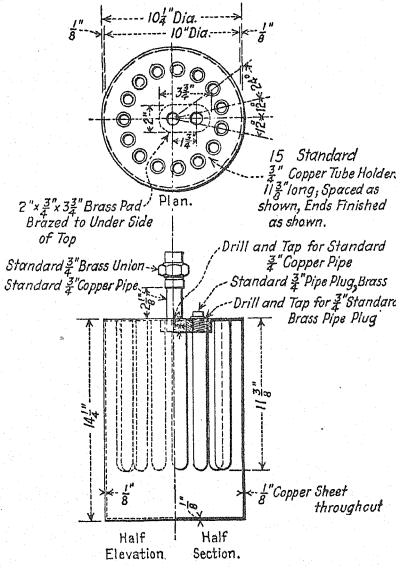


FIG. 5.—Copper Bath for Stability Test.

to allow for expansion and contraction of the air in the tube. Place the tube containing the sample in the heating bath maintained at a temperature of 134.5 ± 0.5 C. The tube shall project not less than 5 nor more than 7 mm. above the top of the bath. Beginning at the end of the first 20 min., inspect the tube at 5-min. intervals by lifting the tube until the methyl violet paper, but not the soluble nitrocellulose, is visible above the surface of the bath. The end point is reached when the test

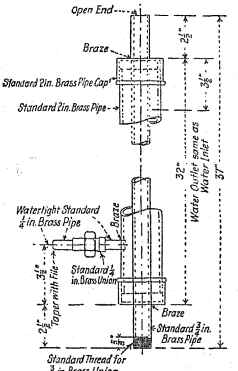


FIG. 6.—Brass Condenser for Stability Test Apparatus.

paper completely loses its violet color. For example, if the color is not completely changed in 20 min. but in completely changed in 25 min. record the stability of the sample as 25 min.

CONSISTENCY

Special Solutions Required

10. The solutions shall conform to the formulas prescribed in Table I.

Apparatus

11. The apparatus shall consist of the following:

(*a*) *Cylinder.*—A glass cylinder 1.0 ± 0.02 in. (25.4 ± 0.5 mm.) in inside diameter, 14 in. in height, and with marks 10.0 ± 0.10 in. (254.00 ± 2.5 mm.) on the side at points about 2 and 12 in. (50.80 and 304.80 mm.) from the top.

(*b*) *Steel Ball.*—A steel ball 0.3120 to 0.3130 in. (0.794 ± 0.001 cm.) in diameter and weighing 2.035 ± 0.010 g.

TABLE I.—SOLUTIONS FOR CONSISTENCY, FILM, AND TOLUOL DILUTION TESTS.

Ingredients	Formula A	Formula B	Formula C	Formula D
Soluble nitrocellulose (dried at 50 C. to constant weight), per cent by weight	12.2	20.0	25.0	12.2
Ethyl acetate,[a] per cent by weight	17.5	16.0	15.0	....
Denatured alcohol, completely denatured (No. 5, 188–190 proof), per cent by weight	22.0	20.0	18.75	....
Toluol,[b] per cent by weight	48.3	44.0	41.25	....
Normal butyl acetate,[c] per cent by weight	....	....	....	87.8

[a] Ethyl acetate conforming to the Standard Specifications for Ethyl Acetate (85 to 88 per cent Grade) (A.S.T.M. Designation: D 302) of the American Society for Testing Materials.[5]

[b] Noncorrosive toluol, 2 C. boiling range, including the boiling point of toluene, 110.7 C.

[c] Normal butyl acetate conforming to the Standard Specifications for Normal Butyl Acetate (88 to 92 per cent Grade) (A.S.T.M. Designation: D 303) of the American Society for Testing Materials.[5]

Procedure

12. (*a*) Consistency is determined by putting the soluble nitrocellulose in solution using a standard formula and noting the rate at which a standard steel ball drops through the solution. Formula A, B, or C (Table I) is usually used for determining consistency. Use formula A unless the sample gives a viscosity with it of 6 sec. or less, in which case use formula B; if below 3 sec. in formula B use formula C. The solution of the nitrocellulose will be

completed somewhat more quickly if the alcohol and toluol are added first and the mixture then allowed to stand 5 or 10 min. before the ethyl acetate is added. Completely dissolve the sample in the solvent mixture by agitating in a tightly-closed container. Fill the glass cylinder with this solution. Stopper the cylinder, allow to stand until all air bubbles have passed out of the solution, and bring to a temperature of 25.0 ± 0.1 C. Place the cylinder in a vertical position and release the steel ball at the center of the upper surface of the solution and allow it to fall through the solution. Record as the consistency of the sample the number of seconds required for the ball to pass through the 10-in. column of solution between the 2 and 12-in. (50.80 and 304.80 mm.) marks on the cylinder.

(*b*) *Report.*—In reporting the consistency prefix the letter A, B, or C to the value of the consistency determined in Paragraph (*a*) in order to indicate the solution formula used.

SOLUBILITY AND APPEARANCE OF SOLUTION

Procedure

13. Compare either formula A, B, C, or D (Note) with a freshly-prepared standard solution of the same type of soluble nitrocellulose of the same formula. Make the comparison in small vials and note the color, turbidity, "grain," and "flock."

NOTE.—Formula A, B, or C which was prepared for the consistency determination is ordinarily used. When the soluble nitrocellulose is to be used for purposes requiring extreme transparency and clarity, use formula D.

FILM TEST

Procedure

14. Dilute formula A, B, or C (Table I) with an equal volume of normal butyl acetate and pour the solution beside a freshly-prepared standard solution of the same type of soluble nitrocellulose and of the same formula, on a clean glass plate. Allow to dry in a nearly vertical position in a dust-free atmosphere. Compare the sample with the standard for undissolved particles, which indicate unnitrated cotton or impurities, and for poor flow and poor gloss.

TOLUOL DILUTION

Procedure

15. To 50 ml. of formula D (Table I) in a stoppered bottle, add c.p. toluol (Note) in small quantities from a burette, shaking well after each addition. The first permanent separation of soluble nitrocellulose is taken as the dilution value and is expressed as a percentage by volume of formula D.

NOTE.—Large quantities of butyl acetate and toluol should be reserved for this test to avoid possible variation between different lots.

Standard Methods of

SAMPLING AND TESTING LACQUER SOLVENTS AND DILUENTS[1]

A.S.T.M. Designation: D 268 – 44

ADOPTED, 1933; REVISED, 1941, 1942, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 268; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods cover procedures for the sampling and testing of solvents and diluents for use in the manufacture of nitrocellulose lacquer.

Sampling

2. (*a*) The method of sampling specified in Paragraph (*b*) or (*c*) shall be used, according to the special conditions that obtain.

(*b*) *From Loaded Tank Car or Other Large Vessel.*—The composite sample taken shall be not less than ½ gal. and should consist of small samples of not more than 1 qt. each, taken from near the top and bottom by means of a metal or glass container with removable stopper or top. This device, attached to a suitable pole, shall be lowered to the desired depth, when the stopper or top shall be removed and the container allowed to fill.

(*c*) *From Barrels and Drums.*—At least 5 per cent of the packages in any shipment shall be represented in the sample. The purchaser may increase the percentage of packages to be sampled at his discretion, and it is recommended that every package be sampled in the case of expensive solvents that are bought in small quantity. A portion shall be withdrawn from about the center of each package sampled by means of a "thief" or other sampling device. The composite sample thus obtained shall be not less than 1 qt. and shall consist of equal portions of not less than ½ pt. from each package sampled.

SPECIFIC GRAVITY

Procedure

3. Determine the specific gravity by any convenient method that is accurate to the third decimal point, the temperature of both sample and water being 20 C.

COLOR

Procedure

4. (*a*) Compare the sample and the reference standard, agreed upon by the purchaser and the seller, in 100-ml.

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these methods were published as tentative from 1927 to 1933, being revised in 1929, 1930, and 1931.

A "tentative revision" of this standard appears in the Tentative Revisions of Standards section at the back of this book.

Nessler tubes having colorless bottoms and having the sides covered with opaque paper for the purpose of eliminating sidelight. Fill the tubes to a height of approximately 24.5 cm., and view them from a point directly above the tube, using a white background beneath the bottom of the tube. For a solvent to be rated water-white, the visible color shall not be darker than a solution of 0.0030 g. of $K_2Cr_2O_7$ in 1 liter of water when tested in the above manner.

(*b*) *Color of Tricresyl Phosphate.*—The Hazen platinum-cobalt solution, prepared as follows,[3] shall be used as the standard for the color test of tricresyl phosphate:

Dissolve 1.246 g. of potassium platinic chloride ($PtCl_4 \cdot 2KCl$) containing 0.05 g. of platinum and 1 g. of crystallized cobalt chloride ($CoCl_2 \cdot 6H_2O$) containing 0.25 g. of cobalt in 100 ml. of HCl (sp. gr. 1.19) and dilute to 1 liter with distilled water. This solution has a standard color of 500.

By placing 4 ml. of the color solution in a colorless 100-ml. Nessler tube and diluting to the mark with distilled water, a standard color of 20 is obtained. For a standard color of 100, 20 ml. of the color solution shall be used.

DISTILLATION

Procedure

5. Conduct the distillation test in accordance with the Standard Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (A.S.T.M. Designation: D 86) of the American Society for Testing Materials,[4] with the following exceptions:

(*1*) The thermometer used shall be an A.S.T.M. Partial Immersion Thermometer graduated in Centigrade degrees having a range of either −20 to +150 C. or −5 to +300 C., according to the boiling range of the material tested, and shall conform to the requirements for these thermometers as prescribed in the Standard Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1) of the American Society for Testing Materials.[4]

(*2*) The temperature at the end point of the distillation, which shall be termed the "dry point," is the temperature observed on the distillation thermometer when the last drop of liquid leaves the bottom of the distillation flask.

(*3*) Make observations of volumes of distillate at the temperature specified for the individual solvent or diluent under test.

NONVOLATILE MATTER

Procedure

6. Place 100 ml. of the sample in a weighed porcelain evaporating dish, evaporate almost to dryness on a steam bath, then heat to constant weight in an oven at 100 to 110 C. The increase in the weight of the dish is the nonvolatile matter of the sample, which should be expressed in grams per 100 ml.

RESIDUAL ODOR

Procedure

7. Determine residual odor for all solvents and diluents in which residual odor is an important property. Dip strips of heavy filter paper, of the same size and shape, to the same depth in beakers or wide-mouth bottles containing the sample and the reference standard. Allow the filter papers to dry in air at room temperature and examine them at suitable intervals for difference in odor.

[3] This method is described in "Standard Methods of Chemical Analyses," Vol. 2, Third edition, Scott.

[4] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

WATER

Procedure

8. Transfer 5 ml. of the sample to a 100-ml. glass-stoppered cylinder, and add 60° A.P.I. gasoline in 5-ml. portions until the total volume specified is obtained, shaking well after each addition. Both sample and gasoline shall be at a temperature of 20 C. when used. Water is indicated by turbidity. If turbidity develops, test the reference standard in the same way and compare with the sample.

ACIDITY

Procedure

9. Using a pipette, transfer 50 ml. of the sample to a small Erlenmeyer flask and titrate with 0.1 *N* KOH in 99 per cent methyl alcohol, using phenolphthalein as an indicator. Calculate the weight of the sample from the specific gravity and report the acidity as milligrams of KOH per gram of sample; in the case of esters also express the results as a percentage by weight of the appropriate acid.

ALKALINITY

Procedure

10. (*a*) Determine the alkalinity of solvents and diluents only when indicated by the results obtained in Section 9. Using a pipette, transfer 50 ml. of the sample to a small Erlenmeyer flask and titrate with 0.1 *N* H_2SO_4, using methyl orange as an indicator. Calculate the weight of the sample from its specific gravity and report the alkalinity as milligrams of KOH per gram of sample.

(*b*) *Alkalinity of Acetone.*—To 100 ml. of acetone contained in a small Erlenmeyer flask, add 0.3 ml. of 0.01 *N* H_2SO_4 and 0.5 ml. of 0.3 per cent aqueous para-nitrophenol. The reaction shall not be alkaline to para-nitrophenol.

ESTER VALUE

Procedure

11. (*a*) Weigh 1 to 2 g. of the sample in an ampoule by first weighing the empty ampoule, warming and filling, and then sealing-off and reweighing (Note 1). Place the ampoule in a 200-ml. Erlenmeyer flask which contains approximately one and one-half times the theoretical quantity of 0.5 *N* aqueous KOH (Note 2) required for complete saponification. If the sample is insoluble in aqueous KOH, add sufficient water-soluble alcohol, free from saponifiable material, to effect solution. Break the ampoule with a stirring rod and connect the flask to a reflux condenser. Heat the flask on a steam bath for 1 to 4 hr., depending on the ester being tested, or until saponification is complete. During the heating, shake the set-up and contents frequently, taking the usual precautions to lose none of the contents. After the apparatus has cooled, rinse the condenser with distilled water and add three drops of phenolphthalein to the contents of the flask as an indicator. Titrate the contents of the flask with 0.5 *N* HCl. Run two blanks along with the sample, using aqueous KOH and the same amount of alcohol as was used with the sample. These blanks should check to the first decimal point. Report the result as percentage of ester by weight, allowing in the calculations for acidity or alkalinity as determined in Sections 9 and 10.

(*b*) *Optional Method.*—Weigh 1 to 2 g. of the sample in an ampoule by first weighing the empty ampoule, warming and filling, and then sealing-off and reweighing (Note 1). Place the ampoule in a pressure bottle of approximately 325-ml. capacity (the spring top magnesium-citrate bottle is satisfactory) which contains one and one-half times

the theoretical quantity of 0.5 *N* aqueous KOH (Note 2) required for complete saponification. If the sample is insoluble in aqueous KOH, add sufficient water-soluble alcohol, free from saponifiable material, to effect solution. Break the ampoule with a glass rod, subsequently washing the liquid adhering to the rod back into the bottle. After thorough agitation, heat the bottle and contents on a steam bath for 1 to 4 hr., depending on the ester being tested, or until saponification is complete. After the proper heating, allow the bottle to cool, then open the bottle, and rinse the under side of the stopper and the sides of the neck with distilled water. Add three drops of the phenolphthalein solution to the contents of the flask as an indicator and titrate with 0.5 *N* HCl. Run two blanks along with the sample, using aqueous KOH and the same amount of alcohol as was used with the sample. These blanks should check, on titration, to the first decimal point. Report the result as percentage of ester by weight, making allowance in the calculations for acidity or alkalinity as determined in Sections 9 and 10.

NOTE 1.—An optional method of weighing the sample is in a small weighing bottle, removing the stopper by means of a glass rod after introduction into the pressure bottle or by agitating the contents of the pressure bottle.

NOTE 2.—Any suitable aqueous alkali may be used in place of aqueous KOH.

COPPER CORROSION TEST

Apparatus

12. The apparatus shall consist of the following:

(*a*) *Flask.*—A 300-ml. flask of chemically-resistant glass with flat bottom and vial mouth.

(*b*) *Glass Condenser.*—A 12-in. glass condenser with the diameter of the condenser tube not less than $\frac{3}{8}$ in. A cork stopper to connect the flask with the condenser.

(*c*) *Copper Strip.*—A copper strip $2\frac{1}{2}$ by $\frac{1}{4}$ in. made from No. 20 B. & S. gage, cold-rolled, polished copper, approximate thickness 0.032 in. and having a $\frac{1}{16}$-in. hole, $\frac{1}{8}$ in. from one end. A piece of soft copper wire about 6 in. in length.

(*d*) *Water Bath.*—A water bath of convenient size and design.

Procedure

13. Polish both sides of the entire copper strip, edges excepted, with a No. 00 emery cloth immediately before using, to avoid discoloration by the atmosphere. Fasten a 6-in. length of soft copper wire through the hole provided near one end of the strip, taking care not to touch the strip with the fingers after polishing. Place the strip in the flask and add 200 ml. of the material to be tested. The material must not contain separated water; filter through a dry filter paper, if necessary, to remove water. Connect the flask to the vertical reflux condenser by means of a properly bored cork stopper. It is absolutely necessary that a cork, not rubber, stopper be used, in order to avoid contamination of the material by sulfur from rubber stoppers. The copper wire may be allowed to extend into the condenser tube for convenience in removing the strip. The strip shall be completely immersed and should preferably lie flat and touch the flask only at the ends of the strip. Place the flask in the water bath, which should be just gently boiling, and immerse the flask to the liquid line of the material within the flask. Remove the copper strip 30 min. from the time the flask was immersed in the bath. Do not touch the copper strip, but remove it by the wire which has been provided. Do not allow the strip to come in contact with separated water during any part of the test, since water causes bad local staining of the copper. If it is desired to preserve the strip for future reference,

dip it immediately in white shellac or lacquer. The sheared edges of the test strip shall not be used as a basis for judgment; flat surfaces only shall be considered.

MERCURY CORROSION TEST

Procedure

14. Shake a 10-ml. sample of the solvent with 1 ml. of clean mercury in a stoppered test tube, and allow to stand 15 min. The mercury shall show no discoloration when compared with a blank run with distilled water.

SULFUR

Procedure

15. *Sulfur (Hydrogen Sulfide and Sulfur Dioxide) in Benzols, Toluols, and Similar Products.*—Make a qualitative test for hydrogen sulfide and sulfur dioxide at the time of performing the distillation test by hanging strips of moistened lead acetate paper and starch iodate paper on the end of the condenser tube. If the lead acetate paper shows discoloration, H_2S is present but not SO_2. In this case both papers will usually be discolored. If the lead acetate paper shows no discoloration but the starch iodate paper develops a blue color, SO_2 is present but not H_2S. If neither paper shows discoloration, neither H_2S nor SO_2 is present.

DIMETHYLKETONE

Reagents

16. (*a*) *Sodium Thiosulfate (0.05 N).*—Prepare a 0.05 *N* solution of $Na_2S_2O_3$ and standardize carefully against $K_2Cr_2O_7$. For accurate work, standardized burettes shall be used and burette corrections and temperature corrections applied. The 50-ml. burettes with National Bureau of Standards certificates are recommended, both for the standardization of solutions and for the actual determination of dimethylketone.

(*b*) *Iodine (0.1 N).*—Prepare and standardize a 0.1 *N* iodine solution at the time of making a determination. Standardize by titrating against the 0.05 *N* $Na_2S_2O_3$, 50 ml. of iodine mixed with the same amounts of NaOH and H_2SO_4 as are used in the determination of dimethylketone. Make the titrations in triplicate and use the average of the three with burette and temperature corrections as the blank in making the calculations.

(*c*) *Sodium Hydroxide (1 N).*—Prepare 1 *N* NaOH and standardize against 2 *N* H_2SO_4.

(*d*) *Sulfuric Acid (2 N).*—Prepare 2 *N* H_2SO_4 and check against 1 *N* NaOH; adjust the strength so that 25 ml. of the acid will neutralize 50 ml. of the NaOH.

(*e*) *Starch Solution.*—Stir 2 to 3 g. of potato starch or 5 g. of soluble starch with 100 ml. of 1 per cent salicylic acid solution, add 300 to 400 ml. of boiling water, and boil the mixture until the starch is practically dissolved. Dilute to 1 liter.

Preparation of Sample

17. Carefully weigh approximately 1.4 g. of acetone by one of the following methods and dilute to 1 liter:

(*a*) Blow glass ampoules $\frac{5}{8}$ to $\frac{3}{4}$ in. in diameter, fill them with acetone, and seal. Determine the exact weight of the sample by the difference in weight of the empty and filled ampoule. Reject samples varying in weight by more than plus or minus 0.1 g. from 1.4 g. Drop the glass ampoules into 500-ml. glass-stoppered bottles containing 200 ml. of boiled distilled water and break the ampoules by shaking. Carefully crush the capillary stem with a glass rod to allow thorough washing out of the acetone, and dilute the sample and combined washings to 1 liter at 20 C.

(*b*) A quick easy method is to weigh

out 1.75 ml. of acetone from a 2-ml. pipette graduated in 0.1 ml. into a small glass-stoppered weighing bottle, conical shape, capacity 12 to 15 ml. This volume will weigh approximately 1.4 g. Invert the weighing bottle and hold the mouth under the surface of freshly boiled distilled water in a 1-liter beaker. Remove the stopper under water by means of a glass rod with a hook on the end. Thoroughly wash out the bottle, transfer the water and acetone to a 1-liter flask, and dilute to the mark at 20 C.

Procedure

18. Pipette three 25-ml. aliquots from each of three flasks prepared in accordance with Section 17(*a*) or (*b*) into separate 750-ml. Erlenmeyer flasks containing 50 ml. each of the 1 *N* NaOH solution. Into each flask, while shaking constantly, run 50 ml. of the 0.1 *N* iodine. Stopper the flasks and allow to stand at about 20 C. (cooling necessary in warm weather) for at least 20 min. At the end of this time, pour 26 ml. of the 2 *N* H_2SO_4 into the first flask and titrate immediately against 0.05 *N* $Na_2S_2O_3$. The solution must be acid before any $Na_2S_2O_3$ is added; otherwise, the determination will be ruined. When the color of the iodine has almost faded out, add starch solution and continue the titration until the blue color disappears and the bright yellow color of the iodoform suspension is reached. Read the milliliters of $Na_2S_2O_3$, apply the burette and temperature corrections, and subtract the corrected reading from the average corrected blank. Calculate the percentage of dimethylketone (pure acetone) by weight from the following formula:

$$A = \frac{(b - s) \times N \times 0.9675 \times 40}{W}$$

where:

A = percentage of dimethylketone by weight,
b = milliliters of $Na_2S_2O_3$ for titration of blank,
s = milliliters of $Na_2S_2O_3$ for titration of sample,
N = normality of $Na_2S_2O_3$ and
W = weight of acetone taken for dilution.

NOTE.—The following precautions shall be observed:

1. Only boiled distilled water shall be used in preparing the solutions and throughout the determination.

2. Standardized glassware shall be used and burette and temperature corrections shall be applied to all readings.

3. Weight of sample taken and amount of iodine solution used shall be as specified. With these amounts, the 35 to 40 per cent excess of iodine, found to be optimum for completion of the iodoform reaction will be present. Use of the small glass-stoppered bottle described in Section 17(*b*) with the 2-ml. pipette insures uniformity in the weight of sample taken.

4. The iodine solution shall be added slowly and while shaking constantly. The National Bureau of Standards calibrated burette with fine tip and delivery time of approximately 2 min. has been found very satisfactory.

5. A slight excess of acid shall be added just before the titration with thiosulfate. Too great an excess will give low results.

POTASSIUM PERMANGANATE TEST

Procedure

19. (*a*) *For Acetone.*—To 100 ml. of acetone contained in a 250-ml. glass-stoppered container and maintained at 25 C., add while stirring slightly, 1 ml. of 0.1 per cent $KMnO_4$ solution. Conduct this test in the dark as sunlight appreciably affects the results.

(*b*) *For Tricresyl Phosphate.*—Shake a 10-g. sample vigorously for 2 min. with 50 ml. of a 0.01 *N* freshly prepared solution of $KMnO_4$ in distilled water and let stand for 30 min.

FLASH POINT

Procedure

20. Determine the flash point in

accordance with the Standard Method of Test for Flash Point by Means of the Tag Closed Tester (A.S.T.M. Designation: D 56) of the American Society for Testing Materials.[4]

VOLATILE MATTER IN TRICRESYL PHOSPHATE

Procedure

21. Place a 25-g. sample of the tricresyl phosphate in a weighed porcelain evaporating dish and heat for 3 hr. in an oven at 100 to 105 C. The loss in weight of the dish and contents is the volatile matter of the sample and shall be expressed as a percentage of the original sample.

FREE PHENOLS IN TRICRESYL PHOSPHATE

Reagents

22. (*a*) *Sodium Hydroxide Solution (2.5 per cent).*

(*b*) *Sodium Bromide-Bromate Solution* containing 10.09 g. of sodium bromide and 2.95 g. of sodium bromate per liter.

(*c*) *Potassium Iodide Solution (6 per cent).*

(*d*) *Hydrochloric Acid (sp. gr. 1.19).*

(*e*) *Starch Solution (1 per cent).*

(*f*) *Sodium Thiosulfate (0.0394 N).*

Procedure

23. Weigh a 10-g. sample of the tricresyl phosphate and transfer to a 250-ml. volumetric flask; add 50 ml. of NaOH (2.5 per cent) at 65 C. and shake the flask vigorously for 3 min. Dilute this solution to the 250-ml. mark and filter. Pipette a 100-ml. portion of the filtrate into a 300-ml. flask for titration. To the flask add from a burette 15 ml. of the sodium bromide-bromate solution and then 5 ml. of HCl (sp. gr. 1.19). Add the HCl quickly and put the stopper in place immediately. Shake the flask and allow to stand for 15 min. Add 10 ml. of the potassium iodide solution and titrate the solution with the 0.0394 *N* sodium thiosulfate solution, using the starch solution as an indicator. Add the starch solution near the end of the titration. The fading out of the blue color marks the end point of the titration. The reactions are as follows:

$$5NaBr + NaBrO_3 + 6HCl = 6NaCl + 3H_2O + 3Br_2$$

$$CH_3C_6H_4OH + 3Br_2 = CH_3C_6HBr_3OH + 3HBr$$

Run a blank titration as described above, using the same reagents.

Calculation

24. Calculate the percentage of free phenols (cresol) as follows:

$$C = \frac{(a - b) \times 0.00071 \times 100 \times 2.5}{W}$$

where:

C = free phenols (cresol) per cent,
a = milliliters of $Na_2S_2O_3$ for titration of the blank,
b = milliliters of $Na_2S_2O_3$ for titration of the sample, and
W = weight of sample (10 g.).

TOLUENE (TOLUOL) DILUTION RATIO

Definition

25. *Toluene (Toluol) Dilution Ratio* of a solvent is the maximum number of unit volumes of toluene that can be added to a unit volume of the solvent to just cause persistent heterogeneity at a concentration of 8 g. of nitrocellulose per 100 ml. of combined solvent and toluene.

Reagents

26. (*a*) *Toluene (Toluol)* conforming to the Standard Specifications for Industrial Pure Toluene (Toluol) (A.S.T.M. Designation: D 362) of the American Society for Testing Materials.[4]

(*b*) *Nitrocellulose* conforming to the Standard Specifications and Tests for Soluble Nitrocellulose (A.S.T.M Designation: D 301) of the American Society for Testing Materials,[4] and in addition of such quality that, when used in determining the toluene dilution ratios of normal butyl acetate and methyl normal propyl ketone, it will give results between the following limits:

	Toluene Dilution Ratio
Normal butyl acetate[a]	2.73 to 2.83
Methyl normal propyl ketone[b]	3.80 to 3.90

[a] The normal butyl acetate, corrected to 90 per cent ester, shall conform to the Standard Specifications for Normal Butyl Acetate (A.S.T.M. Designation: D 303) of the American Society for Testing Materials.[4]

[b] The methyl normal propyl ketone shall have a degree of purity of over 99 per cent as determined by the hydroxylamine method.

Important Factors in Conducting Test

27. (*a*) *Concentration of Nitrocellulose at End Point.*—The dilution ratio increases as the solids content of nitrocellulose at the end point becomes less. It is therefore necessary to set an arbitrary solids content as part of the definition of dilution ratio, and for this purpose a solids content of 8 g. per 100 ml. of solvent plus toluene has been adopted.

(*b*) *Character of End Point.*—The end point shall be taken as the first evidence of heterogeneity in the solution. In the case of a gel end point, a time period of 2 min. shall be allowed for the gel structure to form.

(*c*) *Temperature of Test.*—The test shall be carried out at a temperature of 25 ± 1 C.

Procedure

28. (*a*) *Drying of Nitrocellulose.*—Every precaution shall be exercised in the drying of nitrocellulose which shall be dried by blowing with hot air at a temperature under 75 C. until constant weight within 1 per cent is obtained. Care shall be exercised that the temperature does not exceed 75 C. It is preferable to keep the air at approximately 65 C. in order to be safe.

The material shall be removed from the drying apparatus immediately after blowing as an explosion may occur when the material is not removed promptly after the air has been turned off. Glass shall not be used for the drying tube because of the danger of building up a static charge.

(*b*) *Preparation of Solution.*—The probable toluene dilution ratio of the solvent shall first be estimated in order to make up the initial nitrocellulose solution in the proper concentration (Note). This may be done by referring to published data on similar materials or by a preliminary determination. A solution shall then be prepared using 5 ± 0.01 g. of dry nitrocellulose in the amount of solvent as prescribed in Table I.

TABLE I.

Probable Toluene Dilution Ratio	Volume of Solvent per 5 g. of Nitrocellulose at 25 ± 3 C.
1	25.0 ml.
2	16.7 ml.
3	12.5 ml.
4	10.0 ml.
5	8.3 ml.

The nitrocellulose shall be weighed into a 125-ml. cork-stoppered, tared, Erlenmeyer flask, or other suitable container, on an analytical balance, and then the volume of solvent indicated in the above table shall be added from a burette to the nitrocellulose in the flask. Care shall be taken to prevent loss of solvent by evaporation. The stoppered flask shall be shaken or allowed to stand until the nitrocellulose is completely dissolved before starting titration with the toluene. Care shall be taken not to wet the stopper with the solution during the early stages of the titration. When a high concentration of nitrocellulose in solvent is required, dispersion may be more quickly accomplished

by adding a portion of the toluene to the solvent, which reduces the solids concentration and thus lowers the viscosity of the solution and increases the rate of dispersion.

NOTE.—This procedure for preparation of the solution is specified in order to avoid running a large number of titrations to arrive at a solids content of 8 g. of nitrocellulose per 100 ml. of combined solvent and toluene at the end point.

min. shall elapse between the additions of toluene as the end point is approached.

The initial concentration determined from Table I has been selected to give approximately 10 g. of nitrocellulose per 100 ml. of volatiles at the first end point. An additional quantity of solvent shall now be added and the titration continued to the final end point,

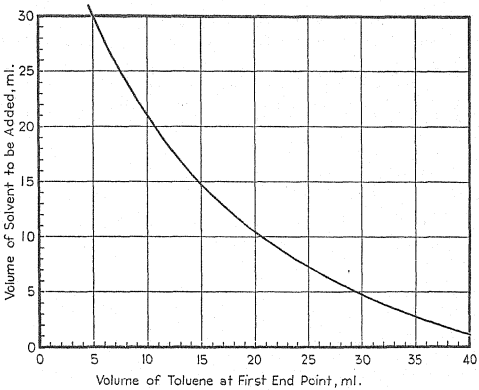


FIG. 1.—Volume of Solvent to be Added to Complete Titration *versus* Volume of Toluene Used to Reach First End Point.

(*c*) *Titration.*—The toluene maintained at 25 ± 3 C. shall be titrated into the flask in small additions, the increments being not over 0.5 ml. as the end point is approached. After each addition, the flask shall be stoppered and vigorously shaken to disperse any gel or precipitate thrown down by local over-concentration of toluene. The end of the titration shall be considered to have been reached when the first definite evidence of heterogeneity persists at 25 ± 1 C. With solvents that give a gel end point, a period of 2

which shall be reached at a solids concentration of 8 ± 1 g. per 100 ml. of volatile matter. The correct amount of solvent to add to accomplish this depends on the amount of toluene that was required in the preliminary titration. The curve of Fig. 1 is so constructed as to make the volume of solvent to be added direct-reading when the volume of toluene used to reach the first end point is known. After the addition of the volume of solvent read from the curve in Fig. 1, the titration shall be completed to the final end point.

Calculation[5]

29. (*a*) The dilution ratios and solids concentrations at both the preliminary and the final end points shall be calculated as follows:

$$\text{Dilution ratio} = \frac{\text{vol. of toluene}}{\text{vol. of solvent}}$$

$$\text{Solids concentration per 100 ml. of volatile matter} = \frac{\text{wt. of nitrocellulose}}{\text{vol. of solvent} - \text{vol. of toluene}}$$

(*b*) A graph shall now be constructed having dilution ratios for the ordinate and solids concentration for the abscissa, and the two sets of values calculated as above shall be plotted as two points on the graph. One of the points shall be very close to 8 g. per 100 ml. of volatile matter. The correct value at exactly 8 g. per 100 ml. of volatile matter may be interpolated by drawing a straight line connecting the two experimentally determined points. While the curve showing relationship between dilution ratio and solids concentration is not necessarily a straight line, the error made by interpolating or extrapolating from the straight line connecting the two points is negligible in the proximity of 8 ± 1 g. per 100 ml. of volatile matter.

[5] Due to the influence of possible variations in the composition of commercial solvents on the resulting toluene dilution ratio, it is recommended that if data obtained by this method is to be published, the composition of the material under test be stated and the character of the end point be indicated.

Standard Method of Test for

ELONGATION OF ATTACHED LACQUER COATINGS WITH THE CONICAL MANDREL TEST APPARATUS[1]

A.S.T.M. Designation: D 522 - 41

ADOPTED, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 522; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test is intended for determining the elongation of attached lacquer coatings when applied to flat sheet metal of uniform surface texture.

Apparatus

2. The conical mandrel test assembly[3] illustrated in Fig. 1 shall be used.

Metal Specimens

3. (*a*) The base metal (Note) of the specimen upon which the lacquer coating is applied shall be cold-rolled steel conforming to the chemical requirements of S.A.E. steel[4] No. 1010, dead soft, finish A, $\frac{1}{32}$ in. in thickness. The steel used shall conform to the dimensional requirements of the Standard Specifications for Cold-Rolled Strip Steel (A.S.T.M. Designation: A 109) of the American Society for Testing Materials.[5] The dimensions of the base metal shall be $4\frac{1}{2}$ in. in width, with a maximum length of $7\frac{1}{2}$ in. and a maximum thickness of $\frac{1}{32}$ in. The surface preparation of the base metal shall be agreed upon by the purchaser and the seller. Prior to application of the finish those edges of the specimen which will be bent in the test shall be rounded slightly to remove burrs in order to eliminate anomalous effects associated with cracking of a sharp metallic edge when bent over the small end of the mandrel.

NOTE.—Other suitable base materials for the test specimen may be employed. The apparatus is designed for testing metal panels having a maximum thickness of $\frac{1}{32}$ in. The method for calculating the elongation of lacquers applied to base materials other than the steel specified in Section 3 is contained in the Appendix.

(*b*) The lacquer whose elongation is to be determined shall be applied to the clean base metal panels in such a manner that a coating of uniform thickness[6] is obtained. The time between the application of the lacquer and the testing of

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, this method was published as tentative from 1939 to 1941.

[3] Blueprints of detailed drawings for the construction of this apparatus are available at a nominal cost from the Headquarters of the American Society for Testing materials, 260 S. Broad St., Philadelphia 2, Pa.

[4] 1944 S.A.E. Handbook, p. 303.

[5] 1944 Book of A.S.T.M. Standards, Part I.

[6] See papers by P. H. Walker and J. G. Thompson on "Some Physical Properties of Paints," *Proceedings*, Am. Soc. Testing Mats., Vol. 22, Part II, p. 464 (1922); and by H. G. Arlt, Bell Laboratories *Record*, Vol. XIV, No. 7, p. 216 (1936).

the specimen, as well as the temperature and humidity environment used for maturing the lacquer film during this period, shall be agreed upon by the purchaser and the seller.

Conditioning and Number of Tests

4. At least three duplicate specimens shall be tested under conditions of temperature and humidity mutually agreed upon by the purchaser and the seller. If it is desirable to check elongation values at another time or place, the film thickness and the temperature and humidity conditions at the time of test shall be the same as those at which the original determinations were made. Unless otherwise specified by the purchaser and the seller, elongation measurements shall be made at 77 ± 2 F. and 50 ± 5 per cent relative humidity after equilibrating to these conditions.

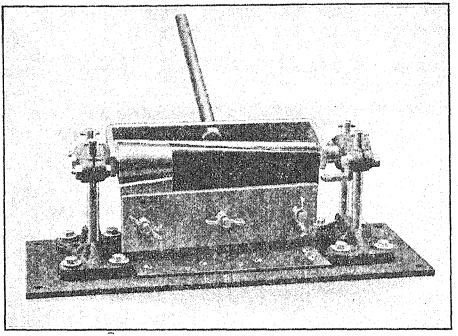


FIG. 1.—Conical Mandrel Test Apparatus.

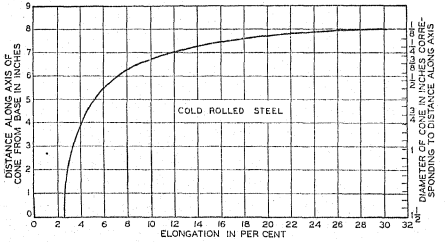


FIG. 2.—Distance Along Cone and Corresponding Mandrel Size *versus* Percentage Elongation for Specimens on Cold-Rolled Steel $\frac{1}{32}$ in. in Thickness.

Procedure

5. With the operating lever of the apparatus in a horizontal position, the test specimen shall be slipped between the mandrel and the drawbar with the

finish side towards the drawbar and rigidly clamped in a vertical position adjacent to the mandrel by placing the long edge behind the clamping bar in such a manner that the panel is always set up to the narrow end of the mandrel. Two sheets of No. 1 brown Kraft wrapping paper, substance 30, thoroughly lubricated on each side with talc, shall be slipped between the specimen and the drawbar and held in position only by the pressure of the drawbar against the paper. The lever shall then be moved at uniform velocity to bend the specimen approximately 180 deg. in about 15 sec. Care shall be taken at the end of the stroke so as not to unnecessarily deform the specimen. The bent surface of the specimen shall be examined immediately with the unaided eye for cracking. Having determined and suitably marked the end of the first continuous crack starting from the small end of the mandrel, which shall be considered as the end point, the drawbar shall then be brought to the starting position and the panel removed from the mandrel. The elongation of the finish shall then be determined from the plotted curve in Fig. 2. This curve represents the relation between the percentage of elongation and the diameter of the conical mandrel. The relation between the distance along the conical mandrel and the corresponding diameter has also been plotted on this curve.

NOTE: *Example.*—Suppose a visual examination of the finish on the bent cold-rolled steel specimens $\frac{3}{32}$ in. in thickness shows that the end of the first crack in the coating is at a distance of 5 in. from the base of the cone. It is merely necessary to refer to Fig. 2 and to read directly the percentage of elongation of the film at the designated distance from the base of the cone. A correction for thickness of coating shall be added to the elongation as determined from Fig. 3. If the film thickness were 0.002 in., from Fig. 2 the elongation at 5 in. from the base of the cone is 5.2 per cent. From Fig. 3 the correction to be added per 0.001 in. of coating is 0.305 per cent at 5 in. from the base of the cone. Thus, when the total film thickness is 0.002 in., the actual percentage elongation at 5 in. is 5.2 + 2 (0.305) which equals 5.8 per cent. The film thickness of the coating shall be determined either with a calibrated dial, a microscope scratch method, or any other suitable method.

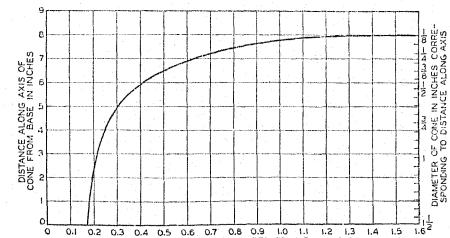


FIG. 3.—Correction for Thickness of Film.

(See Appendix, p. 1066)

APPENDIX

Assuming elastic conditions of the base material (which, of course, is not the case but is useful in establishing a limiting condition), the theoretical elongation for each mandrel size can be calculated. Under this assumption the moduli of elasticity for tension and compression are equal in magnitude and the neutral plane (that is, the plane where the bent bar is neither elongated nor compressed) is at the centroidal axis of the specimen. In a bar having a thickness, t, bent around a mandrel of radius, r, the neutral plane would be located at a distance $t/2$ from the surface of the bar, and the perimeter of the bent section along the neutral axis, assuming the specimen to be bent through 180 deg., is equal to $\pi\ (r + t/2)$. At the upper surface of the bar, the perimeter would be equal to $\pi(r + t)$, and the elongation of the bar at the surface would be:

$$\text{Elongation} = \pi(r + t) - \pi(r + t/2)$$

$$\text{Elongation} = \pi(t/2)$$

and the percentage elongation at the surface would be:

$$\text{Elongation, per cent} = \frac{\pi\, t/2 \times 100}{\pi(r + t/2)}$$

$$\text{Elongation, per cent} = \frac{t}{2r + t} \times 100$$

Using this formula, the theoretical elongation (under elastic conditions) at the surface of the specimen can be calculated for any thickness of specimen and mandrel size, resulting in the following values for three specimen thicknesses and six mandrel sizes:

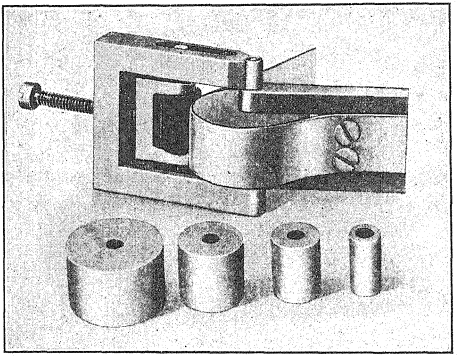


FIG. 4.—Jig for Determining Elongation of Bent Metal Specimens.

Specimen Thickness, in.	Elongation, per cent					
	Mandrel Size = 2 r					
	1 in.	¾ in.	½ in.	⅜ in.	¼ in.	⅛ in.
t = 1/64	1.5	2.0	3.0	4.0	5.9	11 1
t = 1/32	3.0	4.0	5.9	7.7	11.1	20 0
t = 1/16	5.9	7.7	11.1	14.3	20.0	33.3

Actually the specimens are elongated considerably past their elastic limit, so that the elongations obtained with the mandrel test are

considerably higher than the values calculated above. Since it is impossible to calculate the position of the neutral plane when the elastic limit has been exceeded, a special jig should be used to measure the elongation directly.

This jig (Fig. 4) consists of two forks for holding the mandrel and specimen in position and a roller with which the specimen is tightly wrapped around and held against the mandrel.

Method of Measuring Elongation with Jig.—Two parallel gage scratches shall be made with a sharp stylus on the edge of the specimen and so located that when it is wrapped around the mandrel the included angle between the scratches will be approximately 180 deg. In locating the scratches, it is necessary to make sure that they will be within the section of the specimen which after bending is tightly wrapped against the mandrel. Having located the scratches, the linear distance between them shall be measured to within 0.0002 cm.[7] The specimen shall then be clamped in the jig and the roller brought down tight against it. The roller shall then be drawn around causing the specimen to be tightly wrapped and secured against the surface of the mandrel. In order to hold the jig in this position the forks shall be held together with a C-clamp. The clamped jig shall then be rigidly mounted on the table of the measuring machine.[7] Each of the gage scratches shall then be made to coincide with the vertical cross hair of the measuring eyepiece while the horizontal cross hair is tangent to the curvature of the upper surface of the specimen. The angle reading shall be taken at each of these points. The difference between the two angle readings is a measure of the included angle formed by the two gage scratches and the radii drawn coincident with the scratches. The angle readings shall be read in minutes and seconds to a precision of plus or minus 5 sec. The total diameter of the mandrel and the bent specimen shall also be determined. From these data the percentage elongation at the surface of the specimen may be calculated as follows:

$$L = \frac{a\pi}{180} r$$

$$\text{Elongation, per cent} = \frac{L - d}{d} \times 100$$

where:

a = angle in degrees,
L = arc length,
r = radius of curvature for system (mandrel radius + t), and
d = original distance between scratches.

[7] A star comparator is suitable for this purpose.

Standard Methods of

TESTING NITROCELLULOSE CLEAR LACQUERS AND LACQUER ENAMELS[1]

A.S.T.M. Designation: D 333 – 40

ADOPTED, 1937; REVISED, 1940.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 333; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods cover procedures for testing nitrocellulose clear lacquers and lacquer enamels.

Weight per Gallon

2. (*a*) The weight in pounds per U. S. gallon shall be obtained by multiplying the specific gravity by 8.32. The specific gravity shall be determined by any convenient method that is accurate to three significant figures, the temperature of both sample and water being 20 C.

(*b*) *Mobile Liquids.*—For sufficiently mobile liquids, a convenient apparatus is an accurately calibrated volumetric flask.

(*c*) *Viscous Materials.*—For very viscous materials which cannot be conveniently introduced into a volumetric flask, any convenient vessel of known capacity may be used. A suitable apparatus is described in the Standard Method of Test for Specific Gravity of Road Oils, Road Tars, Asphalt Cements, and Soft Tar Pitches (A.S.T.M. Designation: D 70) of the American Society for Testing Materials.[3]

Drying Time

3. The sample and a mutually agreed upon reference standard shall be flowed, without thinning, crosswise on a clean 4 by 8-in. glass plate, in a location free from draft, and examined for drying at specified periods such as 3 min., 15 15 min., 1 hr., and 24 hr. The drying shall be judged by touching or pressing the films with the finger at approximately the same points.

Gloss

4. A panel for the gloss test shall be prepared by pouring the sample and a mutually agreed upon reference standard on an opaque, mutually agreed upon surface in such a manner that the two materials flow together along an adjacent edge. The panel shall be examined for gloss

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these methods were published as tentative from 1931 to 1937, being revised in 1932, 1933, and 1934.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

after 24 hr. A convenient method of determining apparent gloss is to hold the test panel at an angle of incidence approaching 180 deg. to a source of artificial light and to note the comparative sharpness of the definition of the image (for example, the lamp filament).

Homogeneity

5. The sample shall be thinned with the thinner recommended, in the proportions recommended. The thinned sample shall be flowed on a glass panel and dried in a dust-free atmosphere. The panel shall be examined by reflected and transmitted light for pigment dispersion and freedom from particles of foreign matter.

Print Test

6. (*a*) The test panel for the print test shall be prepared in a manner agreed upon by the purchaser and the seller. The test panel shall be covered with two pieces of ordinary cheesecloth over which shall be placed a piece of felt about $\frac{3}{32}$ in. in thickness. A known uniform pressure shall then be applied to the felt over a circular area of at least 1 in. in diameter for a given length of time. The time allowed for the lacquer to dry before applying the pressure shall also be mutually agreed upon.

(*b*) Tests shall be made under definite, mutually agreed upon conditions of temperature and humidity, or, if this is not possible, a test on a reference standard shall be made at the same time.

Outdoor Exposure

7. At least two test panels shall be exposed outdoors on an exposure rack, in full sunlight, at an angle of 45 deg. from the vertical, facing south, and examined at regular intervals for color, gloss, etc., and compared with a reference standard panel prepared and exposed in a manner agreed upon by the purchaser and the seller.

Nonvolatile Matter

8. (*a*) Into a flat-bottom metal or glass dish containing a stirring rod, 4 to 6 g. of the sample to be tested shall be weighed in such a manner as to minimize the loss of volatile solvents. Then 100 ml. of acetone shall be added and stirred until solution of the material is complete, and 10 ml. of water added slowly, while stirring vigorously. The solution shall be evaporated to dryness over a hot water bath and then heated for 1 hr. in an oven at 100 to 105 C., allowed to cool 20 min. in a desiccator, and weighed. (The dish should be covered in such a manner as to allow solvents to escape and exclude contamination by dirt in the atmosphere.) If the precipitate or residue is lumpy, it shall be redissolved in the solvent combination, and precipitated again with water.

(*b*) The method of determining nonvolatile matter in varnishes, as described in Sections 7 and 8 of the Standard Methods of Testing Oleoresinous Varnishes (A.S.T.M. Designation: D 154) of the American Society for Testing Materials,[3] may be used if agreed upon by the purchaser and the seller, with the additional requirement that the sample shall be reheated and reweighed until the weight is constant to within 0.001 g.

Consistency

9. (*a*) *Apparatus.*—The consistency cup shall be so designed as to deliver 50 ml. of the sample in from 30 to 100 sec. A suitable design is shown in Fig. 1.

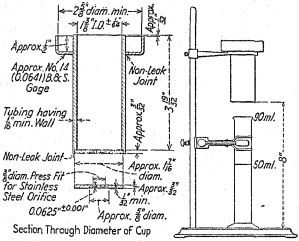


FIG. 1.—Consistency Cups and Apparatus Assembly for Testing Lacquers and Lacquer Enamels.

NOTE 1.—Four consistency cups constitute a set, as follows:

	Diameter of Orifice, in.
First cup	0.07 ± 0.0001
Second cup	0.10 ± 0.0001
Third cup	0.15 ± 0.0001
Fourth cup	0.25 ± 0.0001

NOTE 2.—Cups made of brass or bronze. Orifice disks made of 18 per cent chromium, 8 per cent nickel stainless steel. The diameter of the orifice pressed in the cup shall be stamped on the cup.

NOTE 3.—Fractional dimensions subject to permissible variations of plus or minus 0.01 in., unless otherwise specified.

(*b*) *Procedure.*—The sample to be tested and the consistency cup shall be brought to a temperature of 25 C. (preferably by means of a thermostat). The consistency cup shall then be mounted in the clamp provided for the purpose and the receiving cylinder placed in position. With the outlet of the cup closed by means of the finger, the sample shall be poured into the cup until it is filled to overflowing. The excess shall then be struck off with a straight edge, the finger over the outlet removed and the sample allowed to flow into the receiving cylinder. The number of seconds from the time the finger is removed from the orifice until the top of the meniscus reaches the 50-ml. mark on the cylinder shall be determined by a stop watch and shall be recorded as the consistency of the material.

Standard Methods of

TESTING SOLUBLE NITROCELLULOSE BASE SOLUTIONS[1]

A.S.T.M. Designation: D 365 – 39

ADOPTED, 1939.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 365; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods cover the procedures for testing of soluble nitrocellulose base solutions which are made by dispersing various kinds and concentrations of soluble nitrocellulose in various solvent mixtures.

Basis of Purchase

2. Since the desired specifications and compositions of soluble nitrocellulose base solutions vary greatly, the limits desired for each particular type shall be agreed upon by the purchaser and the seller.

Sampli g

3. Samples shall be taken from not less than 10 per cent of each lot in the shipment. These samples shall then be composited to represent each lot or batch.

CONSISTENCY (VISCOSITY)

Consistency Tests

4. (*a*) *For Consistencies of 3 to 500 sec.*—The falling-ball consistency test apparatus and procedure described in Sections 10 to 12 of the Standard Specifications and Tests for Soluble Nitrocellulose (A.S.T.M. Designation: D 301) of the American Society for Testing Materials[3] shall be used to determine the consistency of those solutions having a consistency from 3 to 500 sec. when tested in that apparatus.

(*b*) *For Consistencies Less than 3 sec.*—The consistency test apparatus and procedure described in Section 9 of the Standard Methods of Testing Nitrocellulose Clear Lacquers and Lacquer Enamels (A.S.T.M. Designation: D 333) of the American Society for Testing Materials[3] shall be used to determine the consistency of those solutions having a consistency of less than 3 sec. when tested in the falling-ball apparatus referred to in Paragraph (*a*).

(*c*) *For Consistencies Over 500 sec.*—The apparatus and procedure described in Sections 5 and 6 shall be used to determine the consistency of those solutions having a consistency greater than

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these methods were published as tentative from 1933 to 1939, being revised in 1939.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

500 sec. when tested in the falling-ball apparatus referred to in Paragraph (*a*).

Apparatus

5. The consistency test apparatus, shown in Fig. 1, shall consist of the following:

(*a*) *Glass Tube.*—A glass tube (preferably resistant glass),[4] 2 ± $\frac{1}{32}$ in. (50.8 ± 1.0 mm.) in inside diameter and 10 in. in length, with marks 5 ± $\frac{1}{16}$ in. (177 ± 1 mm.) apart, the upper one being 3 in. (76.2 mm.) from the top of the tube.

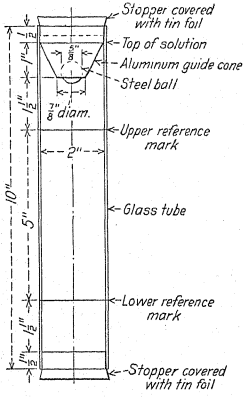


FIG. 1.—Apparatus for Consistency (Viscosity) Test of Solutions Having Consistencies over 500 sec.

(*b*) *Steel Ball.*—A $\frac{5}{8}$-in. steel ball, 0.625 ± 0.001 in. (15.88 ± 0.02 mm.) in diameter, and weighing 16.536 ± 0.10 g.

(*c*) *Aluminum Guide Cone.*—A guide cone of light gage aluminum (approximately 0.02 in. in thickness) as shown in Fig. 1. The orifice of the guide cone shall be $\frac{7}{8}$ in. in diameter, the conical portion 1 in. in height, the cylindrical portion $\frac{1}{2}$ in. in height, and the outside diameter shall be slightly under 2 in. so as to fit snugly into the viscosity tube.

(*d*) *Stoppers.*—Stoppers made preferably of rubber and covered with tin foil.

Procedure

6. (*a*) The tube shall be filled in any convenient manner whereby bubbles do not form and no appreciable amount of solvent is lost. Among other methods this can be done by immersing the lower end of the open tube in the solution and applying suction at the upper end of the tube. In this manner the tube can be filled in from 5 to 10 sec. without the introduction of air bubbles or an appreciable loss of solvent. The bottom of the tube shall then be closed with a stopper covered with tin foil. The aluminum guide cone shall be pushed slowly into the top of the tube and a stopper covered with tin foil inserted into the top of the guide.

(*b*) The tube and its contents shall be brought to a temperature of 25 ± 0.1 C. by placing in a suitable bath. At least 30 min. shall be allowed for the solution to reach temperature equilibrium. For accurate measurements the tube shall be kept during the determination either in a thermostat or suspended within a considerably larger cylinder of water at the specified temperature.

(*c*) The upper stopper shall be removed only long enough to place the ball in the center of the tube; this can be done conveniently with crucible tongs. The principal value of the guide cone is to retard the ball sufficiently at the start of its fall so that the solution will close over the ball and not leave on the top of the ball a large "trailer bubble." If the guide is not used a big trailer bubble usually accompanies the ball, retarding its fall and, if the bubble is off center on

[4] Pyrex glass is very satisfactory for this purpose.

the ball, pulling the latter away from the center of the tube.

(*d*) The time of fall in seconds shall be measured from the instant the bottom of the ball is level with the upper reference mark on the tube until it reaches the lower mark on the tube.

NOTE.—The steel ball can be removed (in order to leave the same material in the tube for a check run) by removing the lower stopper. However, a small air bubble is usually introduced in this way. It is preferable to invert the tube, removing the guide to get the ball out. It is often necessary to put a few drops of solvent in the guide lip to loosen it from the tube on account of the solution drying at the edge of the tube. When the latter method is used for removing the ball, a larger bubble traverses the tube than when the former method is used, but a large bubble moves sufficiently fast, even in a very viscous solution, to escape at the top in a few minutes, whereas small bubbles take hours to escape.

NONVOLATILE MATTER

Procedure

7. The percentage of nonvolatile matter shall be determined in accordance with the procedure described in Section 8 of the Standard Methods of Testing Nitrocellulose Clear Lacquers and Lacquer Enamels (A.S.T.M. Designation: D 333) of the American Society for Testing Materials.[3]

APPEARANCE OF SOLUTION

Procedure

8. The appearance of the soluble nitrocellulose base solution shall be compared with a reference standard agreed upon by the purchaser and the seller (both the reference standard and sample solutions shall be thoroughly agitated before making observations) in similar bottles for turbidity, hair, grain, and insoluble matter.

DEPTH OF COLOR

Apparatus

9. The apparatus used for the preparation of the color standards and for the depth of color determinations shall consist of the following:

(*a*) *Light.*—Source of transmitted light.[5]

(*b*) *Bottles.*—Three dozen 2-oz. screw-cap, square, glass bottles.

(*c*) *Flasks.*—Several 1-liter volumetric flasks.

(*d*) *Burettes.*—Two 50-ml. burettes.

(*e*) *Balance.*—An analytical balance.

(*f*) *Colorimeter.*—Dubosq colorimeter.[5]

(*g*) *Color Glass.*—A piece about 25 mm. square and 10 mm. thick of Corning glass, signal yellow No. 330.

Special Solutions Required

10. (*a*) *Potassium Platinic Chloride* (K_2PtCl_6), c.p.

(*b*) *Cobalt Chloride* ($CoCl_2 \cdot 6H_2O$), c.p.

(*c*) *Hydrochloric Acid* (*sp. gr. 1.19*).

(*d*) *Caramel* (sugar coloring).

(*e*) *Phenol.*

Preparation of Color Standards

11. (*a*) *Platinum - Cobalt Color Standards.*—The platinum - cobalt color standards shall be prepared as follows: Weigh out on an analytical balance exactly 1.246 g. of c.p. potassium platinic chloride (K_2PtCl_6) and 1.000 g. of c.p. crystallized cobalt chloride ($CoCl_2 \cdot 6H_2O$). Dissolve in distilled water, add 100 ml. of HCl (sp. gr. 1.19), and dilute to 1000 ml. with distilled water. This solution is color standard No. 10.[6] Prepare color standards Nos. 1 to 10, inclusive, by accurately measuring from burettes directly into the square 2-oz. bottles the quantities shown in Table I of the No. 10 color standard and distilled water. After preparation of the color standards the bottles shall be

[5] Not absolutely necessary, but will increase accuracy and be more convenient.

[6] This solution has an assigned value of 500 on the Hazen color scale, see *American Chemical Journal*, Vol. XIV, p. 300.

sealed with corks and paraffin before putting on the screw caps. The platinum-cobalt color standards are permanent for approximately six months. The color standards from No. 12 to No. 500, inclusive, shall be made from a caramel solution, but the color is based on the platinum-cobalt color standard.

TABLE I.—PLATINUM-COBALT COLOR STANDARDS.

Platinum-Cobalt Color Standards	Quantity of No. 10 Color Standard, ml.	Quantity of Distilled Water, ml.
No. 1	5	45
No. 2	10	40
No. 3	15	35
No. 4	20	30
No. 5	25	25
No. 6	30	20
No. 7	35	15
No. 8	40	10
No. 9	45	5
No. 10	50	0

(*b*) *Caramel Color Standards.*—The caramel color standards shall be prepared as follows: Dilute caramel (sugar coloring) in the ratio of about 1 ml. of caramel to 100 ml. of distilled water in a glass vessel. Add 0.5 per cent phenol and agitate thoroughly. Adjust the concentration of this solution so that when it is diluted in the ratio of 1 ml. of solution to 49 ml. of distilled water, to which has been added 0.5 per cent phenol, the solution will match color No. 10 of the platinum-cobalt color standard. This caramel solution before dilution is color No. 500 (Note). Prepare color standards No. 12 to No. 500, inclusive, by accurately measuring from burettes directly into the square 2-oz. bottles the quantities shown in Table II of color No. 500 caramel standard and distilled water to which has been added 0.5 per cent phenol. After preparation of the color standards, the bottles shall be sealed with corks and paraffin before putting on the screw caps. All of the caramel color standards are permanent for approximately one month but should be checked semimonthly.

NOTE.—It is more convenient to check the caramel color standard No. 500 against a colored glass which has been previously standardized against the platinum-cobalt color standard; a piece of glass about 25 mm. square by 10 mm. thick of signal yellow No. 330 manufactured by the Corning Glass Works may be standardized and used as a check on the caramel solution. This must be done by use of a colorimeter.

TABLE II.—CARAMEL COLOR STANDARDS.

Caramel Color Standards	Quantity of No. 500 Caramel Color Standard, ml.	Quantity of Distilled Water plus Phenol, ml.
No. 12	1.2	48.8
No. 15	1.5	48.5
No. 20	2.0	48.0
No. 25	2.5	47.5
No. 30	3.0	47.0
No. 35	3.5	46.5
No. 40	4.0	46.0
No. 45	4.5	45.5
No. 50	5.0	45.0
No. 60	6.0	44.0
No. 70	7.0	43.0
No. 75	7.5	42.5
No. 80	8.0	42.0
No. 90	9.0	41.0
No. 100	10.0	40.0
No. 125	12.5	37.5
No. 150	15.0	35.0
No. 175	17.5	32.5
No. 200	20.0	30.0
No. 250	25.0	25.0
No. 300	30.0	20.0
No. 350	35.0	15.0
No. 400	40.0	10.0
No. 450	45.0	5.0
No. 500	50.0	0.0

Procedure

12. A square 2-oz. glass bottle shall be filled with the material to be tested and this shall be matched against one of the color standards using either direct daylight or indirect artificial transmitted light, the latter being preferred. The number of the color standard matched is the depth of color of the material.

Standard Methods of

CHEMICAL ANALYSIS OF WHITE LINSEED OIL PAINTS[1]

A.S.T.M. Designation: D 215 – 41

ADOPTED, 1929; REVISED, 1935, 1937, 1941.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 215; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods cover the procedures for the chemical analysis of the usual white linseed oil paints.

Preparation of Sample

2. On receipt of a sample, make a record of the label, noting especially the brand, the name of the manufacturer, and any statement as to the composition of the paint and the net contents. Weigh the unbroken package, open, note odor and condition of the contents, pour into a clean container, and mix thoroughly by pouring from one container to the other, finally leaving the well-mixed sample in the second container, which shall be tightly closed. The well-mixed sample shall be used at once for the analysis. The original can and cover may be cleaned with gasoline, wiped dry, and then weighed. This weight subtracted from the original weight will give the net weight of the contents. If desired, the specific gravity of the paint may be determined and the weight per gallon calculated, and the volume of paint and the capacity of the container may be measured.

Reagents

3. (*a*) *Extraction Mixture.*—Mix 10 volumes of ether (ethyl ether), 6 volumes of benzol, 4 volumes of methyl alcohol, and 1 volume of acetone.

(*b*) *Aqueous Sodium Hydroxide.*—Dissolve 100 g. of NaOH in distilled water and dilute to 300 ml.

(*c*) *Alcoholic Sodium Hydroxide Solution.*—Dissolve pure NaOH in 95 per cent ethyl alcohol in the proportion of about 22 g. per 1000 ml. Let stand in a stoppered bottle. Decant the clear liquid into another bottle and keep well stoppered. This solution should be colorless or only slightly yellow when used. It will keep colorless longer if the alcohol is previously treated with NaOH (about 80 g. to 1000 ml.), kept at about 50 C. for 15 days, and then distilled.

(*d*) *Wijs Solution.*—Dissolve iodine in glacial acetic acid that has a melting point of 14.7 to 15 C. and is free from reducing impurities, in such proportion

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these methods were published as tentative from 1925 to 1929, being revised in 1927, 1928, and 1929.

that 13 g. of iodine will be present in 1000 ml. of solution. The preparation of the iodine monochloride solution presents no great difficulty, but it shall be done with care and accuracy in order to obtain satisfactory results. There shall be in the solution no sensible excess either of iodine or more particularly of chlorine over that required to form the monochloride. This condition is most satisfactorily attained by dissolving in the whole of the acetic acid to be used the requisite quantity of iodine, using a gentle heat to assist the solution if necessary. Set aside a small portion of this solution while pure and pass dry chlorine into the remainder until the halogen content of the solution is doubled. Ordinarily it will be found that by passing the chlorine into the main part of the solution until the characteristic color of free iodine has just been discharged, there will be a slight excess of chlorine, which is corrected by the addition of the requisite amount of the unchlorinated portion until all free chlorine has been destroyed. A slight excess of iodine does little or no harm, but excess of chlorine must be avoided.

(*e*) *Standard Sodium Thiosulfate Solution.*—Dissolve pure sodium thiosulfate in distilled water (that has been well boiled to free it from carbon dioxide) in the proportion of 24.83 g. of $Na_2S_2O_3 \cdot 5H_2O$ to 1000 ml. of the solution. It is best to let this solution stand for about two weeks before standardizing. Standardize[3] with pure resublimed iodine, pure potassium bi-iodate, or pure KIO_3. This solution will be approximately 0.1 *N*, and it is best to leave it as it is after determining its exact iodine value, rather than to attempt to adjust it to exactly 0.1 *N*. Preserve in a stock bottle provided with a guard tube filled with soda lime.

(*f*) *Starch Solution.*—Stir up 2 to 3 g. of potato starch or 5 g. of soluble starch with 100 ml. of salicylic acid solution (1 per cent), add 300 to 400 ml. of boiling water, and boil the mixture until the starch is practically dissolved, then dilute to 1 liter.

(*g*) *Potassium Iodide Solution.*—Dissolve 150 g. of KI free from KIO_3 in distilled water and dilute to 1000 ml.

(*h*) *Acid Ammonium Acetate Solution.*—Mix 150 ml. of 80 per cent acetic acid, 100 ml. of water, and 95 ml. of NH_4OH (sp. gr. 0.90).

(*i*) *Ammonium Polysulfide.*—Pass H_2S gas into 200 ml. of NH_4OH (sp. gr. 0.90) in a bottle immersed in running water or in iced water until the gas is no longer absorbed; then add 200 ml. of NH_4OH (sp. gr. 0.90) and dilute with water to 1000 ml. Digest this solution with 25 g. of flowers of sulfur for several hours and filter.

(*j*) "*Lead Acid.*"—Mix 300 ml. of H_2SO_4 (sp. gr. 1.84) and 1800 ml. of distilled water. Dissolve 1 g. of c. p. lead acetate in 300 ml. of distilled water and add this to the hot solution, stirring meanwhile. Let stand at least 24 hr. and siphon through a thick asbestos filter.

(*k*) *Potassium Permanganate Solution.*—Dissolve 3.2 g. of pure $KMnO_4$ in 1 liter of distilled water, let stand 8 to 14 days, siphon off the clear solution (or filter through an asbestos filter), and standardize against National Bureau of Standards' standard sample 40*c* of sodium oxalate as follows: In a 400 ml. beaker dissolve 0.25 to 0.30 g. (accurately weighed) of sodium oxalate in 250 ml. of hot water (80 to 90 C.) and add 15 ml. of diluted sulfuric acid (1:1). Titrate at once with the $KMnO_4$ solution, stirring the liquid vigorously and continuously. The $KMnO_4$ must not be added more rapidly than 10 to 15 ml. per min., and the last 0.5 to 1 ml.

[3] Treadwell-Hall, "Analytical Chemistry," Vol. 2.

must be added drop by drop with particular care to allow each drop to be fully decolorized before the next is introduced. The temperature of the solution should not be below 60 C. by the time the end point is reached. (Too rapid cooling may be prevented by allowing the beaker to stand on a small asbestos-covered hot plate during the titration. The use of a small thermometer as a stirring rod is most convenient.) The weight of sodium oxalate used multiplied by 0.833 gives its iron equivalent. The $KMnO_4$ solution should be kept in a glass-stoppered bottle painted black to keep out light.

The iron (Fe) value of the $KMnO_4$ multiplied by 1.076 theoretically equals its antimony (Sb) equivalent. However, for use in determining antimony, the $KMnO_4$ is best standardized as follows: To 0.25 g. of pure metallic antimony in a 500-ml. resistant glass Erlenmeyer flask,[4] add 12 to 15 ml. of H_2SO_4 (sp. gr. 1.84) and 10 to 12 g. of K_2SO_4. Heat until all the antimony is dissolved, cool, dilute to 250 ml. with water, add 20 ml. of HCl (sp. gr. 1.19), cool to 10 to 15 C., and titrate with the $KMnO_4$ solution until a faint pink color is obtained. For special work, after digesting, dilute to 100 ml. with water, add 1 to 2 g. of Na_2SO_3, and boil until all the SO_2 is expelled. This is shown when no blue color is obtained with starch-iodate paper (see below); the volume will be reduced about one-half. Dilute to 250 ml. with water, add 20 ml. of HCl (sp. gr. 1.19), and complete as described.

(*l*) *Standard Potassium Ferrocyanide.*—Dissolve 22 g. of pure $K_4Fe(CN)_6 \cdot 3H_2O$ in water and dilute to 1000 ml. To standardize, transfer about 0.2 g. (accurately weighed) of pure metallic zinc or freshly ignited pure zinc oxide to a 400-ml. beaker. Dissolve in 10 ml. of HCl (sp. gr. 1.19) and 20 ml. of water. Drop in a small piece of litmus paper, add NH_4OH until slightly alkaline, then add HCl until just acid, and then 3 ml. of HCl (sp. gr. 1.19). Dilute to about 250 ml. with hot water and heat nearly to boiling. Run in the $K_4Fe(CN)_6$ solution slowly from a burette, while stirring constantly, until a drop tested on a white porcelain plate with a drop of the uranyl indicator shows a brown tinge after standing 1 min. A blank should be run with the same amounts of reagents and water as in the standardization. The amount of $K_4Fe(CN)_6$ solution required for the blank should be subtracted from the amounts used in standardization and in titration of the sample. The standardization must be made under the same conditions of temperature, volume, and acidity as obtained when the sample is titrated.

(*m*) *Uranyl Indicator for Zinc Titration.*—A solution of uranyl nitrate (5 per cent) in water or a solution of uranyl acetate (5 per cent) in water made slightly acid with acetic acid.

(*n*) *Alkaline Lead Nitrate Solution.*—Into 100 ml. of KOH solution (56 g. in 140 ml. of water) pour a saturated solution of $Pb(NO_3)_2$ (250 g. in 500 ml. of water) until the precipitate ceases to redissolve, stirring constantly while mixing. Let settle, filter through asbestos, and dilute the clear filtrate with an equal volume of water. About three volumes of the $Pb(NO_3)_2$ solution will be required for one volume of the KOH.

(*o*) *Ammoniacal Cadmium Chloride or Zinc Sulfate Solution.*—Dissolve 8 g. of $CdCl_2 \cdot 2H_2O$ in 200 ml. of water and add 200 ml. of NH_4OH (sp. gr. 0.90), or, dissolve 200 g. of $ZnSO_4 \cdot 7H_2O$ in 1080 ml. of water and 920 ml. of NH_4OH (sp. gr. 0.90).

(*p*) *Standard Potassium Iodate Solution.*—Dissolve 3.6 g. of KIO_3 and 39 g. of KI in 1000 ml. of water. For general

[4] Pyrex glass is very satisfactory for this purpose.

work the theoretical sulfur titer of this solution should be used; for special work, the solution may be standardized against like material, such as a lithopone of known sulfide sulfur content. The theoretical titer is based on standard $Na_2C_2O_4$ and is obtained as follows: To 300 ml. of water in a 600-ml. flask, preferably glass stoppered, add 10 ml. of HCl (sp. gr. 1.19) and 1 g. of KI. Cool and add 10 ml. of 0.1 *N* $KMnO_4$ solution which has been standardized against $Na_2C_2O_4$. Swirl gently, stopper, and let stand for 5 min. Titrate the liberated iodine with standard $Na_2S_2O_3$ solution until the color fades. Then add 10 ml. of starch solution and continue the titration until the blue color is destroyed. Repeat the titration, except to substitute 10 ml. of the KIO_3 solution for the $KMnO_4$ solution. Calculate the normality of the KIO_3 solution.

(*q*) *Starch Indicator for Sulfur Titration.*—(1) To 1000 ml. of boiling water, add a cold suspension of 6 g. of starch in 100 ml. of water and boil vigorously for 5 min. Cool the solution, add 6 g. of $ZnCl_2$ dissolved in 50 ml. of cold water, thoroughly mix and set aside for 24 hr. Decant the clear supernatant liquid into a suitable container, add 3 g. of KI, and mix thoroughly. (2, Optional.) Prepare an emulsion of 6 g. of soluble starch in 25 ml. of water, add a solution of 1 g. of NaOH in 10 ml. of water, and stir the solution until it gelatinizes. Dilute to 1000 ml. with water, add 3 g. of KI, and mix thoroughly.

(*r*) *Starch-Iodate Paper.*—Impregnate filter paper with a solution obtained by heating 2 g. of starch with 100 ml. of water, and, after solution, adding 0.2 g. of KIO_3 dissolved in 5 ml. of water.

(*s*) *Standard Iodine Solution for SO_2.*—Place 15 to 20 g. of pure KI in a liter flask, dissolve in as little water as possible, and then add about 6.4 g. of resublimed iodine. Shake until the iodine is all dissolved, dilute to the mark with water, and mix. This solution is approximately 0.05 *N* and is standardized against 0.05 *N* $Na_2S_2O_3$ to obtain its true normality.

(*t*) *Standard Sodium Thiosulfate Solution for SO_2.*—Prepare and standardize as described above, except that 12.42 g. of pure crystallized $Na_2S_2O_3 \cdot 5H_2O$ are used or the 0.1 *N* solution may be diluted with an equal volume of cold CO_2-free water.

Water[5]

4. Mix 100 g. of the sample in a 250-ml. flask with 75 ml. of toluene. Place the flask in an oil bath, connect with condenser, apply heat to the bath, and distill until about 50 ml. of distillate have been collected in a graduate. The temperature in the flask should be then about 105 to 110 C. The number of milliliters of water collected under the toluene in the receiver is the percentage of water in the paint.

Volatile Thinner

5. Weigh accurately from 3 to 5 g. of the sample into a tared flat-bottomed dish about 8 cm. in diameter, spreading the paint over the bottom. Heat at 105 to 110 C. for 1 hr., cool, and weigh. Calculate the loss in weight as percentage of water and volatile thinner, subtract from this the percentage of water,[5] and report the remainder as volatile thinner.

Nature of Thinner

6. (*a*) Transfer about 150 g. of the paint to a 500 ml. flask fitted with a 2-hole cork stopper carrying a spray trap connected with a vertical condenser. Through the other hole in the stopper pass an influx tube for steam. (This

[5] A convenient apparatus or this determination is shown in Fig. 1 (*b*) of the Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (A.S.T.M. Designation: D 95), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

tube should dip below the surface of the paint.) Heat the flask in an oil bath or an air bath at 100 C. and pass through it a current of steam; with the steam still passing through, raise the temperature of the bath to 130 C. Catch the distillate in a small separatory funnel; continue distillation until 300 ml. of water have been condensed. Portions of this water may be drawn from the cock of the separatory funnel from time to time, but care must be taken not to draw out any of the volatile thinner. Let the distillate stand until it separates into two layers, then draw off the water and filter the volatile thinner through a dry filter paper into a dry flask. If the thinner is apparently turpentine, examine the distillate by the methods described in Sections 7 to 11, inclusive, of the Tentative Methods of Sampling and Testing Turpentine (A.S.T.M. Designation: D 233) of the American Society for Testing Materials.[6] If the thinner is a mixture of turpentine and petroleum spirits, an approximate determination of the amount of turpentine may be made by the polymerization test described in Methods D 233. It should be noted that turpentine is slightly soluble in water (about 0.3 to 0.4 ml. per 100 ml. of water).

(*b*) To test for benzol, add a few drops of the distillate to a small quantity of a mixture of HNO_3 (sp. gr. 1.42) and H_2SO_4 (sp. gr. 1.84), and heat cautiously. The characteristic odor of nitrobenzol will be noted if benzol is present.

(*c*) If the thinner is apparently all petroleum spirits, no further examination is necessary.

(*d*) When the amount of turpentine in the thinner is small so that its presence is questionable, it may be detected by placing two drops of the distillate and 2 to 3 ml. of chloroform in a dry test tube and adding one drop of antimony pentachloride. A slow or slight change in color will indicate the absence of turpentine. A rapid change in color to a dark red or purple will indicate the possibility of turpentine. The iodine number for turpentine by the Wijs method under these conditions is approximately 340. An iodine number of 20 or over will give additional proof of the presence of turpentine and enable calculation of the approximate amount.

[6] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Percentage of Pigment

7. Strain a portion of the well-mixed sample through a No. 80 (177-micron) sieve[7] to remove any skins, and weigh accurately about 15 g. of the strained paint in a weighed centrifuge tube. Add 20 to 30 ml. of the extraction mixture (Section 3), mix thoroughly with a glass rod, wash the rod with more of the extraction mixture, and add enough of the reagent to make a total of 60 ml. in the tube. Place the tube in the container of a centrifuge, surround the tube with water, and counterbalance the container of the opposite arm with a similar tube, or a tube with water. Whirl at a moderate speed until well settled. Decant the clear supernatant liquid, repeat the extraction twice with 40 ml. of extraction mixture and once with 40 ml. of ethyl ether. After drawing off the ether, set the tube in a beaker of water at about 80 C. or on top of a warm oven for 10 min., then in an oven at 105 to 110 C. for 2 hr. Cool, weigh, and calculate the percentage of pigment. Grind the pigment to a fine powder, pass through the No. 80 (177-micron) sieve[7] to remove any skins, and preserve in a stoppered bottle.

[7] Detailed requirements for this sieve are given in the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Percentage of Nonvolatile Vehicle

8. Add together the percentages of water, volatile thinner, and pigment, and subtract the sum from 100. Report the remainder as nonvolatile vehicle.

TESTING NONVOLATILE VEHICLE

Preparation of Fatty Acids

9. (*a*) To about 25 g. of the sample in a porcelain casserole, add 15 ml. of aqueous NaOH and 75 ml. of ethyl alcohol, mix, and heat uncovered on a steam bath until all volatile thinner is driven off and saponification is complete. Add 100 ml. of water, boil, add H_2SO_4 (sp. gr. 1.2) (8 to 10 ml. in excess), boil, stir, and transfer to a separatory funnel to which some water has been previously added. Draw off as much as possible of the acid aqueous layer and any insoluble or precipitated matter, wash once with water, then add 50 ml. of water and 50 ml. of ethyl ether. Shake very gently with a whirling action to dissolve the fatty acids in the ether, but not so violently as to form an emulsion. Draw off the aqueous layer and wash the ether layer with one 15-ml. portion of water and then with 5-ml. portions of water until free from sulfuric acid. Then draw off the water layer completely. Transfer the ether solution to a dry flask and add 25 to 50 g. of anhydrous Na_2SO_4. Stopper the flask and let stand with occasional shaking at a temperature below 25 C. until the water is completely removed from the ether solution, which will be shown by the solution becoming perfectly clear above the solid Na_2SO_4. Decant this clear solution, if necessary, through a dry filter paper into a dry 100-ml. Erlenmeyer flask. Pass a rapid current of dry air (pass through a $CaCl_2$ tower) into the mouth of the Erlenmeyer flask and heat to a temperature below 75 C. on a dry hot plate until the ether is entirely driven off. It is important to follow all of the details, since ether generally contains alcohol, and after washing with water always contains water. It is very difficult to remove water and alcohol by evaporation from fatty acids, but the washing of the ether solution and subsequent drying with anhydrous sodium sulfate removes both water and alcohol. Ether, in the absence of water and alcohol, is easily removed from fatty acids by gentle heat. If the pigment settles out rapidly in a sample of the paint on standing so that sufficient vehicle can be poured off, or if sufficient vehicle is obtained by centrifuging the paint, it will be advantageous to saponify this separated vehicle and liberate and prepare the fatty acids as described.

The above method of preparing the fatty acids directly from the material, rather than from the extracted vehicle, is based upon past experience in sometimes obtaining too low results by the latter method. Occasionally, however, trouble is experienced in saponifying the entire material, due to interference of pigment. In such an instance it is permissible to save the extracted vehicle (see Section 7 on Percentage of Pigment), evaporate the organic solvents on a steam bath, and saponify and prepare the fatty acids in the usual manner from this extract. If the iodine number obtained in this manner passes a given specification, no further work is necessary. If the iodine number is low, it will be necessary to repeat the work directly on the entire material.

The fatty acids prepared as above should be kept in a stoppered flask and examined at once.

(*b*) Instead of the method described in Paragraph (*a*) the following procedure may be used, especially with samples that give trouble by the former: To about 50 g. of the sample in a porcelain

casserole, add 30 ml. of aqueous NaOH (30 per cent) and 125 ml. of ethyl alcohol, mix, and evaporate on a steam bath until the residue is dry. Transfer to a 400-ml. beaker and boil with 200 ml. of water, add H_2SO_4 (sp. gr. 1.2) (25 ml. in excess), boil, stir, filter through large coarse paper, and drain. Scrape mass into a flask, shake violently with ether, centrifuge, decant into a separatory funnel, and wash with small amounts of water until free of H_2SO_4. Transfer ether solution to dry flask and let stand over about 40 g. of anhydrous Na_2SO_4 until ether layer is clear. Decant the clear solution through a filter paper into a dry 100-ml. flask. Pass a rapid current of dry air into the mouth of the flask and heat to a temperature below 75 C. on a dry hot plate until the ether is entirely removed. The fatty acids prepared as above should be kept in a stoppered flask and examined at once.

Mineral Oil and Other Unsaponifiable Matter

10. Place 10 drops of the fatty acids (Section 9), in a 50-ml. test tube, add 5 ml. of alcoholic NaOH (Section 3), boil vigorously for 5 min., add 40 ml. of water, and mix. A clear solution indicates that not more than traces of unsaponifiable matter are present.

Iodine Number of Fatty Acids[8]

11. Place a small quantity of the fatty acids (Section 9), in a small weighing burette or beaker. Weigh accurately. Transfer by dropping about 0.15 g. (0.10 to 0.20 g.) into a 500-ml. bottle having a well ground glass stopper, or an Erlenmeyer flask having a specially flanged neck for the iodine test. Reweigh the burette or beaker and determine the amount of sample used. (If desired the sample may be weighed in a small wide-mouthed vial and the vial containing the weighed sample placed in the bottle or flask.) Add 10 ml. of chloroform. Whirl the bottle or flask to dissolve the sample. Add 10 ml. of chloroform to two empty bottles or flasks like that used for the sample. Add to each bottle or flask 25 ml. of the Wijs solution (Section 3) and let stand with occasional shaking for 1 hr. in a dark place at a temperature of from 21 to 23 C. Add 10 ml. of the KI solution (15 per cent) and 100 ml. of water, and titrate with standard sodium thiosulfate solution (Section 3), using starch as indicator. The titrations on the two blank tests should agree within 0.1 ml. From the difference between the average of the blank titrations and the titration on the sample and the iodine value of the thiosulfate solution, calculate the iodine number of the sample tested. (Iodine number is given in centigrams of iodine to 1 g. of sample.)

Rosin

12. (*a*) *Liebermann-Storch Test.*[9]—To about 1 g. of the fatty acids add 15 ml. of acetic anhydride and shake until solution is complete. Pour a few drops of this solution on a white porcelain plate (a crucible cover serves well) and add a drop of H_2SO_4 (sp. gr. 1.53). A fugitive violet color indicates rosin.

(*b*) *Halphen-Hicks Test.*[10]—Test the fatty acids with the Halphen-Hicks reagent as follows:

SOLUTION A.—One part by volume of phenol dissolved in two parts by volume of CCl_4.

SOLUTION B.—One part by volume of bromine dissolved in four parts by volume of CCl_4.

[8] If appreciable amounts of rosin or of unsaponifiable matter are found to be absent in the vehicle of a paint, the iodine number of the fatty acids gives the best indication (though not proof) of the presence of linseed oil. An iodine number of less than 175 (Wijs) for the fatty acids is an indication that the nonvolatile vehicle was not pure linseed oil.

[9] "Chemical Technology and Analysis of Oils, Fats and Waxes," by J. Lewkowitsch, Vol. 1, p. 623 (1921).

[10] *Industrial and Engineering Chemistry*, Vol. 3, p. 86 (1911).

Add 1 to 2 ml. of Solution A to about 1 g. of the fatty acids and pour this mixture into a cavity of an ordinary porcelain color-reaction plate until it just fills the depression. Immediately fill an adjacent cavity with Solution B. Cover the plate with an inverted watch glass and note the color, if any, produced in the former solution by the action of the bromine vapors from Solution B. A decided purple or deep indigo blue color is an indication of the presence of rosin.

ANALYSIS OF PIGMENT

Qualitative Analysis

Qualitative Analysis

13. A complete qualitative analysis, following the well-established methods, should be made and the quantitative scheme modified as required. Add acetic acid slowly to the pigment until all carbonate is decomposed (noting whether any H_2S is evolved), then add a large excess of acid ammonium acetate solution (Section 3). Boil, filter, and test the filtrate for metals other than lead and zinc (especially calcium and barium). The absence of calcium in this filtrate indicates that the extending pigments contain no $CaCO_3$ or $CaSO_4$; the absence of barium indicates that the extending pigments contain no $BaCO_3$.[11] Wash the matter insoluble in acid ammonium acetate solution with another portion of this solution, and finally with hot water. This insoluble matter is dried, ignited, and tested for siliceous matter, $BaSO_4$, and titanium compounds. To test for the latter, place a small amount of the insoluble matter, or of the original sample (about 0.5 g.), in a 250-ml. resistant glass beaker;[4] add 20 ml. of H_2SO_4 (sp. gr. 1.84) and 7 to 8 g. of $(NH_4)_2SO_4$. Mix well, and boil for a few minutes. A residue denotes the presence of silica or siliceous matter. Cool the solution, dilute with 100 ml. of water, heat to boiling, settle, filter, wash with hot sulfuric acid (5 per cent) until free from titanium. The residue may be tested for lead, barium, and silica. Add hydrogen peroxide to a small portion of the filtrate; a clear yellow-orange color indicates the presence of titanium. Boil another portion of the filtrate with metallic tin or zinc; a pale blue to violet coloration indicates titanium. Treat another portion (about 1 g.) of the pigment with 20 ml. of HCl (1:1) and note whether any H_2S is evolved; boil the solution for about 5 min., add about 25 ml. of hot water, filter, and wash with hot water. Render a small portion of the filtrate alkaline with NH_4OH, acidify with HCl, and add a little $BaCl_2$ solution; a white precipitate ($BaSO_4$) indicates the presence of a soluble sulfate. To another portion of the filtrate add a little H_2SO_4; a white precipitate indicates the presence of lead, soluble barium, or both (some $CaSO_4$ may also separate). Filter, wash to remove free acid, and treat the precipitate with a few drops of KI solution; the formation of yellow PbI_2 indicates the presence of lead. The white precipitate may also be treated with H_2S water; the formation of black PbS indicates the presence of lead. To another portion of the original filtrate add NH_4OH until alkaline, render slightly acid with acetic acid, heat to boiling, and add a little $K_2Cr_2O_7$ solution; a yellow or orange-yellow precipitate indicates the presence of lead, soluble barium, or both. To another portion of the original filtrate add a few drops of $K_4Fe(CN)_6$ solution; a white precipitate with a blueish tinge

[11] If the original sample contained $BaCO_3$ and $PbSO_4$, $CaSO_4$, or other soluble sulfate, the soluble Ba will form with the soluble sulfate a precipitate of $BaSO_4$ which will be determined as "insoluble matter." If the sample contained $SrSO_4$ or $SrCO_3$, some $SrSO_4$ may be counted as $BaSO_4$, some Sr will count as soluble barium, and some may be counted as CaO. This element is not separated, as it probably will not be encountered, or will be present as an impurity in the Ba and Ca compounds.

indicates the presence of zinc. Pass into the remaining portion of the original filtrate a current of H_2S for 5 to 10 min., add an equal volume of water, and pass H_2S into the solution for about 5 min. Filter, wash with H_2S water. Digest the precipitate with ammonium polysulfide, filter, acidify the filtrate with HCl, and warm; the presence of antimony is indicated by the separation of an orange colored precipitate. The filtrate from the H_2S precipitate may be tested for barium, calcium, and magnesium in the usual manner.

Quantitative Analysis, Single Pigments

Procedure

14. If the sample is a single pigment, follow the procedure described in the Standard Methods of Chemical Analysis of White Pigments (A.S.T.M. Designation: D 34) of the American Society for Testing Materials[12] for the particular pigment to be tested.

Quantitative Analysis, Mixed or Composite Pigments

Moisture and Other Volatile Matter[13]

15. Place 1 to 2 g. of the pigment in a wide-mouth, short, weighing tube provided with glass stopper. Heat with the stopper removed for 2 hr. at 105 to 110 C. Insert the stopper, cool, and weigh. Calculate the loss in weight as moisture and other volatile matter.

Loss on Ignition

16. Ignite 1 g. of the pigment to constant weight in a porcelain crucible over a Meker burner.[14]

Insoluble Matter

17. (*a*) Moisten 1 g. of the pigment with a few drops of alcohol, cover, add 40 ml. of HCl (1:1), boil gently for 5 to 10 min. Wash the cover, evaporate to dryness, and heat at about 150 C. for 30 min. to 1 hr. to dehydrate the residue. Moisten the residue with 4 ml. of HCl (sp. gr. 1.19), allow to stand a few minutes, dilute with 100 ml. of hot water, boil a few minutes, filter hot through paper, wash with hot water (until washings give no test for lead and chlorine). Ignite the paper and residue in a platinum or porcelain crucible, cool, and weigh total insoluble matter.[11] (The insoluble matter may be filtered off on a Gooch crucible, washed with hot water, dried at 105 C., cooled, and weighed; then ignited, cooled, and weighed, when it is desired to get the loss on ignition (combined water, organic matter, etc.) of the same, or the insoluble matter is not to be further examined.) If the sample contains titanium pigment, practically all of the TiO_2 will be found in the insoluble matter along with $BaSO_4$ and siliceous matter. Should an examination of the insoluble matter be necessary, it is advisable to remove the TiO_2 before proceeding further. The TiO_2 may be removed (or determined on a separate portion) by the method described in Section 34 of the Standard Methods of Chemical Analysis of White Pigments (A.S.T.M. Designation: D 34) of the American Society for Testing Materials.[12] After removing the TiO_2, the residue containing siliceous matter and $BaSO_4$ may be ignited to remove the filter. To determine $BaSO_4$, mix the ignited insoluble matter with about ten times its weight of anhydrous Na_2CO_3 (grinding the mixture in an agate mortar if necessary) and fuse in a covered platinum crucible, heating about 1 hr. Let cool, place crucible and cover

[12] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

[13] On an extracted and dried pigment, this determination is of little value. If the original paint contained gypsum, a part of the combined water of the latter will be driven off in the drying of the extracted pigment and in the "moisture" determination.

[14] This determination may serve as a rough or approximate check in many cases on the CO_2, water, etc.

in a 200-ml. glazed porcelain casserole,[15] add about 100 ml. of water, and heat until the mass is disintegrated. Filter on paper into a 300-ml. glazed porcelain casserole (leaving crucible and cover in the original casserole) and wash the casserole and filter thoroughly with a hot solution of Na_2CO_3 (1 per cent). Place the casserole containing the crucible and cover under the funnel, pierce the filter with a glass rod, and wash the residue into the original casserole by means of a jet of hot water. Wash the paper with hot HCl (1:1) and then with hot water. Remove the crucible and cover. Evaporate the HCl solution to dryness, and heat at about 150 C. for 30 min. to 1 hr. Moisten the residue with about 10 ml. of HCl (sp. gr. 1.19), dilute with 100 ml. of hot water, boil a few minutes, filter hot through paper, and wash thoroughly with hot water. Dilute the filtrate to a volume of 300 ml., bring to boiling, and add, drop by drop, 5 ml. of H_2SO_4 (1:4). Allow to stand in a warm place an hour or so, filter on a weighed Gooch crucible, wash with hot water, ignite, cool, and weigh as $BaSO_4$. Subtract the sum of the percentages of $BaSO_4$ and TiO_2 from the percentage of total insoluble matter and report the result as the percentage of insoluble siliceous matter.[16]

(*b*) To determine silica, acidify the filtrate from the $BaCO_3$ filtration with HCl, boil to expel CO_2, evaporate to dryness, bake to dehydrate the silica, moisten with HCl, dilute with 100 ml. of hot water, boil and filter through the same paper as was used to recover silica from the $BaCO_3$ portion. Wash thoroughly with hot water and proceed as in a silicate analysis.

(*c*) If it is desired to look for magnesium, combine this last filtrate with the filtrate from the final $BaSO_4$ separation and test for Al_2O_3 and MgO in the usual way. To recover MgO that may have dissolved in the procedure for the elimination of the TiO_2, make the filtrate containing the TiO_2 just alkaline with NH_4OH, bring to boiling, filter and wash. The filtrate may be tested for MgO. Any Al_2O_3 present will be precipitated along with the TiO_2. To recover this, ignite and weigh as TiO_2 and Al_2O_3. Deduct for TiO_2 present in the sample; the difference is Al_2O_3.

Total Lead (Antimony)

18. (*a*) Unite the filtrate and washings (total volume 150 to 200 ml.) from the total insoluble matter, pass H_2S into the solution until it is saturated, add an equal volume of water, and again saturate with H_2S. Filter, wash with water containing a little H_2S, dissolve in hot HNO_3 (1:3), washing the paper with hot water. Add 10 to 20 ml. of H_2SO_4 (1:1), evaporate until copious fumes of sulfuric acid are evolved. Cool, add about 75 ml. of water and then about 75 ml. of 95 per cent ethyl alcohol. Stir, let settle, filter on a Gooch crucible, wash with diluted alcohol, dry in an oven at 105 to 110 C.; or, ignite gently in a radiator[17] or muffle, cool, and weigh as $PbSO_4$. Calculate to PbO.[18]

(*b*) If the pigment contains antimony, filter and wash the sulfide precipitate as described in Paragraph (*a*). Wash the precipitate with a fine jet of water from the paper into a porcelain dish or casserole, add 25 ml. of ammonium polysulfide (Section 3), cover the vessel, and warm the mixture at 40 to 60 C. for 10 to 15 min. while stirring frequently.

[15] A casserole is preferable to a beaker, as silica is dissolved from glass when in long contact with a strong sodium carbonate solution.

[16] Any soluble Al_2O_3 (Fe_2O_3) and in most cases MgO, and sometimes some CaO, come from the siliceous pigment used. MgO generally denotes the presence of asbestine

[17] *U. S. Geological Survey Bulletin 700*, p. 33 (1919).

[18] It is not possible to determine the amount of basic lead carbonate and lead sulfate when carbonates or soluble sulfates of other metals, such as calcium, are present. Also, neither basic lead carbonate nor basic lead sulfate are definite compounds.

Wash the cover, filter through the paper used in the first case, and wash with 2 to 3 per cent Na_2S or $(NH_4)_2S$ solution. Discard the filtrate. Dissolve the residue in hot diluted HNO_3 (1:3), and determine the lead as $PbSO_4$, as described in Paragraph (*a*). Or, the original sulfide precipitate may be discarded and the lead determined on a separate portion of the pigment as follows: To 1 g. of the sample in a covered beaker, add 40 ml. of HCl (1:1) and boil gently for 5 to 10 min. Wash off cover and evaporate to dryness. To the residue add sufficient HCl (sp. gr. 1.19) to dissolve the $PbSO_4$ (with pigments containing considerable amounts of $PbSO_4$, it may be necessary to add 15 to 20 ml. of HCl (sp. gr. 1.19)), add about 50 ml. of hot water, boil a few minutes, filter hot through paper, and wash with hot water until washings give no test for lead. (If the sample contains no insoluble matter, omit the filtration.) To the filtrate add 20 ml. of H_2SO_4 (sp. gr. 1.84) and evaporate until dense white fumes of SO_3 are copiously evolved. Allow to cool, but not below 60 C., and then add slowly 50 ml. of water while agitating the solution. Heat to boiling for several minutes in order to insure complete solution of antimony sulfate. Allow the $PbSO_4$ to settle out until the supernatant liquid is clear, not letting the temperature fall below 60 C. If the liquid does not clear quickly it must be heated longer. When clear, pour the solution through a weighed porcelain Gooch crucible with asbestos mat, decanting the solution as completely as possible without allowing more than a very small amount of $PbSO_4$ to go over into the crucible. Now add 10 ml. more of H_2SO_4 (sp. gr. 1.84) to the $PbSO_4$ in the original beaker, and boil for several minutes. Cool, add slowly 30 ml. of water, and again heat to boiling for a few minutes. Allow the solution to cool to about 60 C. and completely transfer the $PbSO_4$ to the Gooch crucible. Wash with "lead acid" (Section 3) to remove soluble sulfates and finally wash free of acid with diluted alcohol (equal parts of ethyl alcohol or denatured alcohol and water). Dry in an oven at 105 to 110 C., or ignite gently in a radiator or muffle. Calculate to PbO, or determine as $PbCrO_4$ as described in Paragraph (*e*).

(*c*) If soluble compounds of barium or calcium are present, $BaSO_4$ and $CaSO_4$ will be included with the $PbSO_4$. If soluble SiO_2 is present, it will also be included with the $PbSO_4$. In such cases, the $PbSO_4$ precipitate after washing with diluted alcohol may be dissolved in acid ammonium acetate (Section 3) and the lead determined as $PbCrO_4$, as described in Paragraph (*e*). For ordinary work, the amount of $BaSO_4$ dissolved by the acetate treatment may be disregarded.

(*d*) If the pigment contains no soluble antimony, barium, or calcium compounds, the lead may be determined directly on the original pigment, as follows: To 1 g. of the sample in a covered beaker, add 25 ml. of HNO_3 (1:1), and boil gently a few minutes. Wash off cover, evaporate to dryness on a steam bath, moisten with HNO_3, add hot water, and heat a few minutes. Filter and wash with hot water until washings are lead-free. Add 10 to 20 ml. of H_2SO_4 (1:1) to the clear solution, evaporate, and determine lead as $PbSO_4$, as described in Paragraph (*a*).

(*e*) In the absence of soluble compounds of antimony, iron, aluminum, and barium, the following procedure may be used: Treat 1 g. of the original pigment with 25 ml. of HNO_3 (1:1) and proceed as above. To the clear solution, diluted to 200 ml., add NH_4OH in slight excess, acidify with acetic acid, and add 4 to 6 ml. more of this acid.

Heat to boiling and add 10 to 15 ml. of a solution of $K_2Cr_2O_7$ (10 per cent). Heat until the yellow precipitate assumes an orange color, let settle and filter on a weighed Gooch crucible. Wash by decantation until the washings are colorless, finally transferring all of the precipitate. Then wash with 95 per cent ethyl alcohol and then with ether. Dry to constant weight at 110 C., cool, and weigh $PbCrO_4$. Calculate to PbO.

Antimony Oxide

19. (*a*) Transfer 0.3 g. of a straight antimony oxide pigment, or 0.5 g. of a mixed pigment, to a 500-ml. resistant glass Erlenmeyer flask,[4] add 15 ml. of water and 25 ml. of HCl (sp. gr. 1.19). Cover with a watch glass, warm on the steam bath 10 to 15 min. to dissolve the antimony oxide, wash off cover, add 250 ml. of water, and 15 ml. of H_2SO_4 (sp. gr. 1.84). Boil 2 min., cool to 10 to 15 C., and titrate to a faint pink tint with 0.1 *N* $KMnO_4$ solution (Section 3). Calculate to Sb_2O_3.

The above procedure gives only the antimony in the *ous* condition. The following method gives the total antimony (*ous* and *ic* forms): Transfer 0.3 g. of a straight antimony oxide pigment, or 0.5 g. of a mixed pigment, to a 500-ml. resistant glass Erlenmeyer flask,[4] add 15 ml. of H_2SO_4 (sp. gr. 1.84), 10 g. of K_2SO_4, and a 9-cm. filter paper (to furnish carbon to act as a reducing agent). Place a funnel in the neck of the flask, and heat until the solution becomes colorless. Cool, wash off the funnel, dilute to 250 ml. with water, add 20 ml. of HCl (sp. gr. 1.19), and boil 2 min. Cool to 10 to 15 C., and titrate to a faint pink tint with 0.1 *N* $KMnO_4$ solution.[19]

(*b*) *Procedure in Presence of Appreciable Amounts of Iron.*—Treat 1 g. of the mixed pigment, or 0.3 g. of a straight antimony oxide pigment, in a covered 250-ml. beaker with 5 ml. of water and 20 ml. of HCl (sp. gr. 1.19). Heat on the steam bath for 15 min., cool, wash off cover, add 3 g. of tartaric acid and 100 ml. of hot water, and digest a few minutes. Filter, catching the filtrate in a 500-ml. resistant glass Erlenmeyer flask.[4] Wash thoroughly with hot water, dilute to 300 ml. with hot water, and pass in H_2S until the precipitation is complete. (If the sample contains no insoluble matter, dissolve directly in a 500-ml. resistant glass Erlenmeyer flask,[4] add tartaric acid, dilute, and pass in H_2S.) Filter, wash with water containing H_2S until free from HCl, return paper and precipitate to the Erlenmeyer flask, add 15 ml. of H_2SO_4 (sp. gr. 1.84) and 10 g. of K_2SO_4, place a funnel in the neck of the flask, and heat until the solution is colorless. Cool, wash off the funnel, dilute to about 250 ml. with water, add 20 ml. of HCl (sp. gr. 1.19), boil for 2 or 3 min., cool to about 10 C., and titrate to a faint pink tint with 0.1 *N* $KMnO_4$ solution (Section 3). Calculate the total antimony to Sb_2O_3.[20]

[19] If the digestion with H_2SO_4 and K_2SO_4 (plus filter paper) is continued after the solution becomes colorless, some of the antimony may be oxidized from the *ous* to the *ic* condition. In such cases, cool, wash off the funnel, dilute to 100 ml. with water, add 1 to 2 g. of Na_2SO_3 and boil until all of the SO_2 is expelled. This is shown when no blue color is obtained with starch-iodate paper (see Section 3); the volume will be reduced about one half. Dilute to 250 ml. with water, add 20 ml. of HCl (sp. gr. 1.19), and boil 2 min.; cool to 10 to 15 C., and titrate to a faint pink tint with 0.1 *N* $KMnO_4$ solution. Calculate total Sb to Sb_2O_3. Subtract the Sb_2O_3 found by the procedure given in the first paragraph under Antimony Oxide from the total Sb_2O_3 and calculate the residual Sb_2O_3 to Sb_2O_4.

[20] If the digestion with H_2SO_4 and K_2SO_4 (plus filter paper) is continued after the solution becomes colorless, some of the antimony may be oxidized from the *ous* to the *ic* condition. In such cases, cool, wash off the funnel, dilute to 100 ml. with water, add 1 to 2 g. of Na_2SO_3 and boil until all of the SO_2 is expelled. This is shown when no blue color is obtained with starch-iodate paper (see Section 3); the volume will be reduced about one half. Dilute to 250 ml. with water, add 20 ml. of HCl (sp. gr. 1.19), and boil 2 min.; cool to 10 to 15 C., and titrate to a faint pink tint with $KMnO_4$ solution (0.1 *N*).

Soluble Barium

20. Boil the combined filtrate and washings, reduced in volume by evaporation if need be, from the PbS precipitate (Section 18) to expel H_2S. Add a slight excess of H_2SO_4 (1:4) over the amount required to precipitate the barium, heat to boiling, let stand on a steam bath about 1 hr., filter on a weighed Gooch crucible, wash with hot water, dry, ignite, cool, and weigh as $BaSO_4$.[11,21] Calculate to BaO.

Aluminum Oxide (Fe_2O_3, TiO_2, P_2O_5)

21. Boil the filtrate from the PbS to expel H_2S, add a few drops of HNO_3, and continue the boiling a few minutes to oxidize any iron that may be present. In case soluble barium was present, use the filtrate from that determination. To the solution containing at least 5 g. of NH_4Cl per 200 ml. of solution, or an equivalent amount of HCl, add a few drops of methyl red (0.2 per cent alcoholic solution) and heat just to boiling. Carefully add diluted NH_4OH drop by drop until the color of the solution changes to a distinct yellow. Boil the solution for 1 to 2 min. and filter at once. Wash the precipitate thoroughly with hot NH_4Cl solution (2 per cent).[22] Ignite the precipitate, cool, and weigh as Al_2O_3.[23]

Total Zinc

22. (*a*) To the combined filtrate and washings from the alumina precipitate (Section 21), add sufficient HN_4Cl to give 5 g. per 100 ml. of solution, and then add 1 g. of ammonium acetate.[24] Make slightly acid with acetic acid and pass in a current of H_2S to saturation. Allow the precipitate to settle completely, filter on paper, and wash with a solution of acetic acid (2 per cent) saturated with H_2S. Transfer the precipitate and filter to the vessel in which the precipitation was effected, add 30 ml. of water and 10 ml. of HCl (sp. gr. 1.19), heat until all zinc is in solution, add 200 ml. of water and a small piece of litmus paper; add NH_4OH (sp. gr. 0.90) until slightly alkaline, make just acid with HCl, then add 3 ml. of HCl (sp. gr. 1.19), heat nearly to boiling, and titrate with standard $K_4Fe(CN)_6$ solution as in standardizing that solution (Section 3).

(*b*) Zinc may be determined directly on the original sample as follows:[25] Weigh acurately about 1 g. (or an amount that will give a burette reading approximately equal to that obtained in the standardization) of the pigment, transfer to a 400-ml. beaker, add 30 ml. of HCl (1:2), boil a few minutes, add 200 ml. of water and a small piece of litmus paper; add strong NH_4OH until slightly alkaline, render just acid with HCl, then add 3 ml. of HCl (sp. gr. 1.19), heat nearly to boiling, and titrate with standard $K_4Fe(CN)_6$ solution as in standardizing that solution (Section 3).

(*c*) When iron is present, total zinc may be determined directly on the original sample as follows.[25] Weigh accurately about 1 g. (or an amount that will give a burette reading approximately equal to that obtained in the standardization) of the pigment, transfer to a 250-ml. beaker, moisten with alcohol, add 30 ml. of HCl (1:2), boil for 2 or 3 min., and add about 100 ml. of water. Add about 2 g. of NH_4Cl, make slightly alkaline with NH_4OH, heat to boiling, let settle on a steam bath, filter into a 400-ml. beaker and wash the

[21] This will include any $BaSO_4$ that may have been dissolved as such. The weighed precipitate should be tested for $CaSO_4$, and if present, it should be removed by treating with hot diluted HCl, filtering, washing, igniting, and again weighing.

[22] For very accurate work, or when the precipitate is large, the precipitate should be dissolved in HCl (1:1) and the precipitation repeated.

[23] This precipitate may also contain Fe_2O_3, TiO_2, and P_2O_5.

[24] F. A. Gooch, "Representative Procedures in Quantitative Chemical Analysis," 1st Edition, p. 107.

[25] If the sample contains antimony, it should be precipitated by H_2S in the hot acid solution, filtered off, washed, and the filtrate neutralized, etc., for zinc. The H_2S precipitate may also contain PbS. If no sulfide separation is made, any cadmium present will be counted as zinc.

residue once with hot water. Remove the 400-ml. beaker and pour diluted HCl on the residue, catching the filtrate therefrom in the 250-ml. beaker, wash a few times with hot water. Add to this filtrate 1 g. of NH_4Cl and make slightly alkaline with NH_4OH, boil, let settle, filter on paper used for first filtration, and wash thoroughly with hot water, catching the filtrate and washings in the 400-ml. beaker containing the first filtrate. Add a small piece of litmus paper, acidify with HCl, add 3 ml. of HCl (sp. gr. 1.19), heat nearly to boiling, and titrate with standard $K_4Fe(CN)_6$ as described in Paragraph (*b*).

(*d*) With pigments containing ZnO and ZnS, the ZnO may be determined as follows: Weigh accurately 1 g. of the pigment, transfer to a 250-ml. beaker, moisten with alcohol, add about 100 ml. of acetic acid (1 to 3 per cent), stir vigorously but do not heat, cover, and let stand for 18 hr., stirring once every 5 min. for the first 30 min. Filter, wash with acetic acid (1 to 3 per cent) followed by water until the washings give no test for zinc with $K_4Fe(CN)_6$ solution. Dilute the clear filtrate to about 200 ml. with water, add 30 ml. of HCl (1:2), and a small piece of litmus paper; add NH_4OH (sp. gr. 0.90) until slightly alkaline, render just acid with HCl, then add 3 ml. of HCl (sp. gr. 1.19), heat nearly to boiling, and titrate with $K_4Fe(CN)_6$ solution as described in Paragraph (*b*). Calculate this result to Zn, subtract from total Zn, and calculate the difference to ZnS. (Any $ZnCO_3$ or $ZnSO_4$ is included in the ZnO.)

Soluble Calcium

23. Heat to boiling the united filtrate and washings, reduced in volume if need be, from the ZnS precipitate (Section 22), add 1 ml. of NH_4OH and an excess of a hot saturated ammonium oxalate solution. Continue the boiling until the precipitate becomes granular; let stand about 1 hr., filter, and wash with hot water. Ignite, cool, and weigh as CaO;[11,26,27] or, place the beaker in which the precipitation was made under the funnel, pierce the apex of the filter with a stirring rod and wash the precipitate into the beaker with hot water, pour warm diluted H_2SO_4 (1:4) through the paper and wash a few times. Add about 30 ml. of diluted H_2SO_4 (1:4), dilute to about 250 ml., heat to 90 C., and titrate at once with standard (0.1 *N*) $KMnO_4$ solution (the temperature of the solution should not be below 60 C. when the end point is reached; see Section 3). Calculate to CaO.[11,26,27] (The Fe value of $KMnO_4 \times 0.502 = CaO$ value.)

Soluble Magnesium

24. Acidify the filtrate from the calcium precipitate with HCl, add 10 ml. of a saturated solution of $Na(NH_4)HPO_4$ and NH_4OH drop by drop, with constant stirring. When the crystalline $(NH_4)MgPO_4$ has formed, add 5 ml. excess of NH_4OH. Allow the solution to stand in a cool place for not less than 4 hr., preferably overnight.[28] Filter and wash with water containing 2.5 per cent of NH_3. Dissolve the precipitate in a small quantity of hot diluted HCl, dilute the solution to about 100 ml. with water, add 1 ml. of a saturated solution of $Na(NH_4)HPO_4$ and NH_4OH drop by drop, with constant stirring, until the precipitate is again formed as described, and then add 5 ml. excess of NH_4OH. Let the precipitate stand in a cool place for not less than 2 hr., filter on a Gooch

[26] Care must be exercised in this washing, as 1000 ml. of boiling water will dissolve over 0.01 g. of CaC_2O_4.

[27] For more accurate work, the CaC_2O_4 precipitate should be ignited, cooled, cautiously moistened with water, redissolved in HCl, and the solution diluted to 100 ml. Then NH_4OH should be added in slight excess, the liquid boiled, and filtered and washed if a precipitate appears. Then reprecipitate the Ca with NH_4OH and $(NH_4)_2C_2O_4$ as above, filter, wash, ignite, cool, and weigh; or, titrate as described.

[28] The less the amount of magnesium present, the longer the precipitate must be allowed to settle.

crucible, wash with water containing 2.5 per cent of NH_3, ignite, cool, and weigh as $Mg_2P_2O_7$.[29] Calculate to MgO.

Carbon Dioxide

25. Use from 1 to 2 g. of the pigment depending upon the probable CO_2 content, following either of the methods described under the determination of carbon dioxide in the Standard Methods of Chemical Analysis of Limestone, Quicklime, and Hydrated Lime (A.S.T.M. Designation: C 25) of the American Society for Testing Materials.[30, 31]

Total Soluble Sulfur Compounds[11]

26. Treat 1 g. of the pigment in a 400-ml. beaker with 10 ml. of water, 10 ml. of HCl (sp. gr. 1.19) saturated with bromine, and 5 g. of NH_4Cl. Digest (covered) on a steam bath for 5 min., dilute with hot water to about 200 ml., boil for 5 min., filter to separate any insoluble matter, and wash thoroughly with hot water. Nearly neutralize the clear solution in a covered beaker with NaOH solution, complete the neutralization with dry Na_2CO_3, and add about 2 g. more of this reagent. Boil 10 to 15 min., wash off cover, let settle, filter, and wash with hot water. Redissolve the precipitate in HCl (1:1), reprecipitate with Na_2CO_3 as above, filter, and wash thoroughly with hot water. Acidify the united filtrates with HCl, adding about 1 ml. in excess. Boil to expel bromine, and to the clear boiling solution add slowly while stirring an excess of a $BaCl_2$ solution (10 per cent). Let stand on a steam bath for at least 1 hr., filter on a weighed Gooch crucible, wash thoroughly with boiling water, dry, ignite at a dull red heat, cool, and weigh as $BaSO_4$. This will include soluble sulfates, SO_3 formed from SO_2, and the SO_3 that is formed from sulfide sulfur.[21]

Soluble Sulfate[11]

27. Treat 1 g. of the pigment with 10 ml. of water and 10 ml. of HCl (sp. gr. 1.19) and 5 g. of NH_4Cl. Boil until H_2S is expelled, adding more HCl (1:1) if necessary. Dilute with hot water to about 200 ml., boil for 5 min., filter to separate any insoluble matter, and wash thoroughly with hot water. Nearly neutralize the clear solution with NaOH solution and make a double precipitation with Na_2CO_3, as in preceding method, finally weighing as $BaSO_4$ as described above.

Sulfide Sulfur[32, 33]

28. Place 0.5 to 1 g. of the pigment in a flask with about 10 g. of "feathered" or mossy zinc, add 50 ml. of water; insert a stopper carrying a separatory funnel and an exit tube. Run in 50 ml. of HCl (sp. gr. 1.19) from the funnel, having previously connected the exit tube to two absorption flasks in series; the first flask contains 100 ml. of alkaline lead nitrate solution (Section 3), the second flask, 50 ml. of the same solution as a safety device. After all of the acid has run into the evolution flask, heat slowly, finally boiling until the first appearance of steam in the first absorption flask. Disconnect, let the PbS settle, filter, wash with cold water, then with hot water until neutral to litmus paper and washings give no test for lead. Dissolve the PbS precipitate in hot, diluted HNO_3 and determine the lead as

[29] If the sample contained manganese, it will be caught in large part with the $Mg_2P_2O_7$. If desired, Mn may be determined by dissolving the $Mg_2P_2O_7$ in HNO_3 and applying the bismuthate method.

[30] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

[31] If the sample is high in sulfide, for example, contains a high percentage of lithopone, grind 1 to 2 g. of the pigment with dry $K_2Cr_2O_7$, transfer to the evolution flask, add 50 ml. of water, and run in H_2SO_4 (1:1) from the separatory funnel. Or, place at the front of the purifying and drying train a tube containing acidified $CuSO_4$ solution, $KMnO_4$ solution, or CrO_3 solution.

[32] Evolution Method of W. G. Scott, "White Paints and Painting Materials," p. 257; see also "Blair, The Chemical Analysis of Iron."

[33] The percentage of sulfide sulfur can be calculated from the percentages of total zinc and zinc soluble in acetic acid (2 to 3 per cent), assuming the sulfide to be ZnS. See Section 22 (*d*) under determination of zinc.

$PbSO_4$. Calculate to S. For very rapid work, the evolved H_2S may be absorbed in an ammoniacal $CdCl_2$ or $ZnSO_4$ solution (Section 3) contained in two flasks connected in series, the contents of the absorption flasks washed into a vessel with cold water and diluted to about 1 liter, acidified with HCl (sp. gr. 1.19), and titrated with standard KIO_3 solution using starch indicator (Section 3).

Sulfur Dioxide[34]

29. Transfer 10 g. of the pigment to a suitable flask, insert a stopper fitted with a separatory funnel and a spray trap delivery tube,[35] and attach the latter to a condenser. Place about 150 ml. of HCl (1:3) in the funnel, the stopcock being closed,[36] connect the other end of the condenser with a delivery tube which passes through a two-hole stopper and extends nearly to the bottom of an absorption flask; through the other hole of the stopper connect a tube or flask to serve as a safety device. Place 25 ml. of 0.05 *N* iodine solution (Section 3) in the absorption flask (dilute with water if necessary) and 20 ml. of KI solution (10 per cent) in the safety tube; fit stopper in the absorption flask. Open the stopcock and allow the acid to slowly enter the flask. Before all of the acid is admitted, force air (washed with NaOH solution) through the top of the separatory funnel (about two bubbles per second in the KI solution). Boil the solution 3 min. with the air passing through, then remove the source of heat and pass air through for 30 min. Disconnect the absorption vessels, wash the KI solution into the iodine solution, and titrate at once with 0.05 *N* $Na_2S_2O_3$ solution, using starch indicator. Run a blank determination in exactly the same manner except for the omission of the pigment. Subtract this figure from the previous one and calculate the final result to SO_2 (1 ml. 0.05 *N* iodine = 0.0016 g. SO_2).

Matter Soluble in Water

30. Transfer 2.5 g. of the pigment to a graduated 250-ml. flask, add 100 ml. of water, and boil for 5 min. Cool to room temperature, dilute to the mark with water, mix, and allow to settle. Filter the supernatant liquid through a dry filter paper and discard the first 20 ml. of the filtrate. Transfer 100 ml. of the clear filtrate to a weighed dish, evaporate to dryness on a steam bath, dry for 1 hr. in an oven at 105 to 110 C., cool, and weigh. Calculate the percentage of water-soluble matter.

NOTE.—The nature of this may be determined by further examination, as the percentages of SO_3 and CaO may be indicative.

Calculations

31. (*a*) The calculation of the component pigments of a mixed or combination pigment may be a somewhat difficult matter. Certain assumptions must be made, depending upon the complexity of the mixed pigment, as to the composition or formulas of component pigments and as to the manner in which the acidic and basic radicals are combined. Add any Al_2O_3 (Fe_2O_3) found in the soluble portion to the siliceous matter and report the sum as "insoluble siliceous matter" unless the soluble Al is high; in this case, an aluminate is probably present, and the Al_2O_3 should be reported as Al_2O_3. If a small amount of soluble Mg is found, it should also be added to the siliceous matter. If the soluble Mg is high, the presence of $MgCO_3$ is indicated, and the MgO is calculated to $MgCO_3$ as pointed out below. The insoluble siliceous matter reported should be based on the weight

[34] This method is not applicable in the presence of sulfides decomposable under the conditions given.

[35] A Knorr CO_2 apparatus is very convenient. In this case, the vertical condenser may be connected with an absorption tower containing the iodine solution, followed by the KI solution in a suitable tube.

[36] To minimize, if not eliminate, any possible oxidation by the air, add about 1 g. (in one piece) of $NaHCO_3$ to the evolution flask, then add the acid directly to the flask, omitting the separatory funnel and the current of air. Boil the solution until about 50 ml. of distillate has passed over.

obtained on drying the total insoluble matter at 105 C. if the combined water contained therein is to be considered.

(*b*) Report TiO_2 as TiO_2, ZnS as ZnS, and $BaSO_4$ as $BaSO_4$. If $CaCO_3$, $CaCO_4$, $BaCO_3$, and $MgCO_3$ are absent, calculate CO_2 to basic carbonate white lead, $(PbCO_3)_2 \cdot Pb(OH)_2$, and soluble SO_3 to $PbSO_4$. Any excess of Pb is calculated to PbO, added to the $PbSO_4$, and the sum reported as basic lead sulfate; or, multiply the sum of $PbSO_4 + PbO$ by 0.058 to obtain the ZnO; add this result to the $PbSO_4 + PbO$ and report as basic sulfate white lead. (The ZnO factor is based on the assumption that the average composition of commercial basic sulfate white lead is: 78.5 per cent $PbSO_4$, 16.0 per cent PbO, and 5.5 per cent ZnO.) Lead oxide (PbO) should not be reported except in the presence of $PbSO_4$ unless the entire analysis is reported in the elementary or oxide form.

(*c*) If the sample contains CO_2 but no soluble SO_3, calculate total Pb to basic carbonate white lead, $(PbCO_3)_2 \cdot Pb(OH)_2$; calculate residual CO_2 to $CaCO_3$, then to $BaCO_3$ and $MgCO_3$ if soluble Ba and Mg should be present in sufficient amounts to indicate the presence of these carbonates. The CO_2 result will be an index of this. A small amount of residual CaO is probably from the siliceous matter and should be added to the insoluble siliceous matter.

(*d*) A small amount of soluble Ba may be from the $CaCO_3$ used or may be due to the solubility of $BaSO_4$, if this compound is present in the original pigment. This Ba may be calculated to $BaSO_4$ and added to the $BaSO_4$ found in the insoluble matter.

(*e*) If the sample contains soluble SO_3 but no CO_2, calculate CaO to $CaSO_4$ or $CaSO_4 \cdot 2H_2O$; residual SO_3 to $PbSO_4$; add residual PbO to $PbSO_4$ and report sum as basic lead sulfate; or, multiply $PbSO_4 + PbO$ by 0.058 and add the result to the $PbSO_4 + PbO$, and report the total as basic sulfate white lead.

(*f*) If the sample contains $CaCO_3$ ($MgCO_3$, $BaCO_3$) and also basic sulfate white lead, or $CaSO_4$ and basic carbonate white lead, or a mixture of these, it is not possible to determine or calculate the amount of $PbCO_3$ or $PbSO_4$ with any degree of certainty.[11, 13] The presence of appreciable amounts of CaO and SO_3 in the water-soluble matter indicates the probable presence of $CaSO_4$ in the original pigment. The following arbitrary calculations may be made: calculate water-soluble SO_3 to $CaSO_4$ or $CaSO_4 \cdot 2H_2O$, subtract this SO_3 from total soluble SO_3 and calculate the remainder to $PbSO_4$; calculate residual CaO to $CaCO_3$, and then residual CO_2 to $(PbCO_3)_2 \cdot Pb(OH)_2$. If there is an excess of CO_2, calculate to $MgCO_3$ or $BaCO_3$, if the amounts of soluble Mg and Ba indicate the probable presence of these carbonates. Add residual PbO to $PbSO_4$ and calculate, as above, to basic sulfate white lead.

(*g*) Report total antimony as Sb_2O_3.

(*h*) Calculate sulfide sulfur to ZnS, subtract the Zn equivalent to the S from the total Zn, then subtract the Zn required for the basic sulfate white lead, and report the remainder as ZnO.

(*i*) Report moisture and other volatile matter, loss on ignition, SO_2, and matter soluble in water directly.

Standard Method of Test for
RELATIVE DRY HIDING POWER OF PAINTS[1]

A.S.T.M. Designation: D 344 – 39

ADOPTED, 1939.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 344; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test is intended for determining whether the relative dry hiding power of a given sample of paint is less than, equal to, or greater than that of a mutually agreed upon standard. An optional procedure is included which permits a quantitative determination of the relative dry hiding powers of two paints when they differ not too greatly in this respect.

Definition

2. Relative dry hiding power of a paint is the ability of that paint to reduce the contrast of a black and white surface to which it is applied and allowed to dry. It is quantitatively expressed in terms of the proportional spreading rate[3] of paint required to produce the same contrast reduction as obtained with the paint chosen as standard.

Apparatus

3. The apparatus shall consist of the following:

(*a*) *Balance.*—Regulation laboratory balance with capacity of 100 g. and sensitivity of 0.1 g.

(*b*) *Syringe.*—Syringe of 10-ml. capacity.

(*c*) *Paint Brush.*—A good grade paint brush, 1½ in. in width.

(*d*) *Container.*—A ½-pt. can or a 250-ml. beaker.

(*e*) *Test Surface.*—A smooth-surfaced paper chart having adjacent black and white areas and coated with a suitable varnish or lacquer so as to render the surface impervious and resistant to paint liquids. The black areas of the board shall have a diffuse reflectance of less than 1 per cent. The white areas shall have a diffuse reflectance of 75 per cent or greater. The white areas of the boards used for the standard paint and the sample shall not differ in brightness by more than 1 per cent. The chart shall have a test area of not less than 1 sq. ft., and any suitable regular geometrical design of the contrasting areas may be used.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, this method was published as tentative from 1932 to 1939, being revised in 1937 and 1939.

[3] See the definition of "Spreading Rate" in the Tentative Definitions of Terms Relating to Paint, Varnish, Lacquer, and Related Products (A.S.T.M. Designation: D 16), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(*f*) *Illumination.*—A reasonable intensity of diffused light, preferably northern skylight or a source approximating northern skylight.

Materials

4. A standard paint mutually agreed upon by the purchaser and the seller.

Procedure

5. (*a*) The weight per gallon of the standard and sample paints must be known and may be determined by any accepted method.

(*b*) Brushouts of the standard paint and the sample paint shall be prepared as described in Paragraph (*c*) at a convenient and suitable spreading rate mutually agreed upon by the purchaser and the seller and preferably lying within the range of 400 to 800 sq. ft. per gal.

(*c*) The standard paint shall be well stirred and the brush dipped into the paint and worked out on a suitable surface. The desired quantity of paint shall be taken up in the syringe, and the syringe and wet brush weighed in the empty container. The paint shall then be spread over the surface of the chart, the syringe returned to the empty container, and the paint brushed out uniformly over the test surface, care being taken to cover only the test area. The empty syringe, wet brush, and container shall again be weighed. The loss of weight represents the weight of paint applied to the test surface. A test chart shall then be prepared from the sample paint in the same manner and at the same spreading rate. Both painted test surfaces shall then be set aside to dry.

(*d*) After the test surfaces are thoroughly dry, they shall be placed side by side against a flat surface. The standard and sample brushouts shall be viewed in juxtaposition under a reasonable intensity of diffused light, preferably northern skylight or a source approximating northern skylight. The observer should view the brushouts from a distance of 5 to 10 ft.

Relative Dry Hiding Power

6. If the apparent contrast existing on the brushout of the sample paint is adjudged equal to or less than that existing on the brushout of the standard paint, the sample paint shall be said to be equal to or better than the standard paint in relative dry hiding power as defined in Section 2.

Optional Procedure

7. (*a*) In many cases it is not sufficient to indicate that the sample paint is better or poorer than the standard, and the amount of difference shall be determined quantitatively. The optional procedure described in Paragraphs (*b*) and (*c*) is recommended for determining quantitative differences in hiding power. It may be used in place of the procedure outlined in Section 5.

(*b*) The sample paint shall be applied to a brushout board as described in Section 5 (*c*) at the spreading rate mutually agreed upon or at a convenient spreading rate. A series of brushouts shall be made from the standard paint. The difference in spreading rate between successive steps in this series should be approximately 15 per cent. The range of spreading rate covered by this series should be sufficient to give contrasts above and below that of the brushout of the sample paint (estimated from the probable difference in relative dry hiding power, if known) or should cover the entire range of spreading rate convenient to apply with the standard paint. The painted test surfaces shall be set aside to dry.

(*c*) Under the conditions of illumination and viewing, as described in Section 5 (*d*), the brushout of the sample paint shall be compared with a series of brush-

outs of the standard paint, numbered, beginning with one, in order of decreasing spreading rate for convenience of handling. It will be found to be equal in contrast to one brushout of the standard, or to be intermediate in contrast between two adjacent standards in the series of standard brushouts. If the sample is found to be between two adjacent standards in contrast, the sample shall be viewed against these standards with the higher and lower standard on either side. For purposes of grading, the contrast interval between the two standards shall be considered as 10 arbitrary units. The position of the sample shall be estimated on this arbitrary scale. Thus, if the sample appears equally distant in contrast from either standard, it will be graded 5 on the arbitrary scale. If nearer the standard of less contrast, it will be graded 6, 7, 8, or 9 as the case might be. The grading shall be recorded in terms of the number of the standard brushout of higher spreading rate followed by a decimal point and the grading of the arbitrary scale. In making this grading it is important that the sample be graded between the two standards with the standard of lesser contrast first on the right and then on the left. The two gradings thus obtained shall be averaged to give the final grading figure.

Calculation

8. The hiding power of the sample of paint shall be calculated as follows:

$$\text{Hiding power} = \frac{(W_1 - W_2)\frac{X}{10} + W_2}{W_3} \times \frac{S_2}{S_1} \times 100$$

where:

W_1 = weight of paint on the brushout of the heavier grading standard,

W_2 = weight of paint on the brushout of the lighter grading standard,

W_3 = weight of paint on the brushout of the sample paint,

X = contrast grading (decimal portion of the recorded grading),

S_1 = weight per gallon of the standard paint, and

S_2 = weight per gallon of the sample paint.

Standard Method of Test for

CONSISTENCY OF EXTERIOR HOUSE PAINTS AND ENAMEL-TYPE PAINTS[1]

A.S.T.M. Designation: D 562 – 44

ADOPTED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 562; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test is intended for determining the consistency of exterior house paints and enamel-type paints.

Consistency

2. For the purpose of this method, consistency is expressed in terms of the weight required to produce the specified speed.

Apparatus

3. The apparatus shall consist of the following:

(*a*) *Viscosimeter.*—A Stormer viscosimeter with the paddle-type rotor as illustrated in Figs. 1 and 2. Intercalibration of any two viscosimeters may be carried out by determining the load in grams required to produce a shearing rate of 200 rpm. with a standard oil having a viscosity of 10 to 15 poises at 25 C.

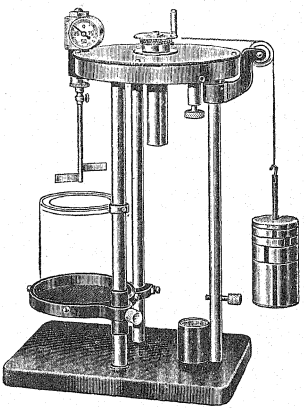


FIG. 1.—Stormer Viscosimeter with Paddle-Type Rotor.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, this method was published as tentative from 1942 to 1944.

(*b*) *Container.*—A 1-pt. friction-top can, $3\frac{3}{4}$ in. in diameter or other suitable container of that diameter having a capacity of 1 pt.

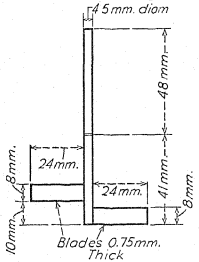


FIG. 2.—Paddle-Type Rotor for Use with Stormer Viscosimeter.

Procedure

4. Thoroughly mix the sample of paint and strain it into the 1-pt. container and allow the paint to stand over 12 to 24 hr. Bring the temperature of the sample to 25 ± 0.25 C. and maintain it at that temperature during the test. Place the container on the platform of the viscosimeter so that the paddle-type rotor is immersed in the sample just to the mark on the shaft of the rotor. Before starting the test, turn the rotor through approximately 100 revolutions in 25 to 35 sec. Then, using different weights determine the time required for 100 revolutions of the rotor. Select weights that will give at least two readings within a range of 27 to 33 sec. Make these determinations from a running start, that is, permit the rotor to make at least 10 revolutions before starting the count of a test.

Report

5. Plot the time in seconds required for 100 revolutions of the rotor against the load in grams. Report the consistency as the weight in grams necessary to produce 100 revolutions of the rotor in 30 sec. This value may be taken from the curve or interpolated from the results obtained.

Standard Method of Test for

SPECTRAL CHARACTERISTICS AND COLOR OF OBJECTS AND MATERIALS[1]

A.S.T.M. Designation: D 307 – 44

ADOPTED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 307; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test is intended to determine the spectral characteristics of light-transmitting objects and of light-reflecting objects and materials, and to designate their colors.

NOTE 1.—The method of designating colors has come into wide acceptance since its recommendation in 1931 by the International Commission on Illumination.[3] It is intended primarily for light-reflecting and light-transmitting specimens whose spectral characteristics have been determined by the spectrophotometer, but it is also applicable to the expression of color measurements by visual or photoelectric colorimeters.

NOTE 2.—If portions of a test specimen to which the measurement applies are homogeneous, the measurement is taken as characteristic of the material of the specimen; thus, a specimen consisting of an opaque paint film or an opaque layer of paper is a specimen of material (paint or paper). But if the effective portions of the specimen consist of two or more materials so combined that the measurement depends upon their arrangement, the measurement characterizes, not a material, but objects which are constructed from the same materials in the same way; thus, a specimen consisting of an incompletely hiding paint film on a ground coat represents, not a material, but simply a group of objects made from the superimposition of a particular finishing coat on a particular ground coat. A specimen of light-reflecting material shall consist of a layer so thick that further increase in thickness will not cause a change in appearance.

Description of Terms and Symbols

2. (*a*) *Tristimulus Designations, X, Y, Z.*—The evaluations of the color as determined in accordance with this method.

NOTE.—The appearance of materials and objects is not completely defined by this evaluation of color, but may also be influenced by other properties such as texture and gloss.

(*b*) *Tristimulus Designations of a Spectrum of Unit Irradiance per Unit Wave Length,* $\bar{x}_\lambda$, $\bar{y}_\lambda$, $\bar{z}_\lambda$.—The functions of wave length which serve to define the *X*, *Y*, and *Z* scales.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, this method was published as tentative from 1929 to 1930, being revised in 1930. It was adopted as standard in 1930, published as standard from 1930 to 1937, but revised and republished as tentative from 1937 to 1939, being revised in 1939. It was again adopted as standard in 1939, published as standard from 1939 to 1942, but revised and republished as tentative in 1942.

[3] *Proceedings*, Eighth Session, Commission Internationale de l'Éclairage, Cambridge, England, September, 1931, pp. 19-29.

NOTE.—The function, $\bar{y}_\lambda$, is known as spectral luminosity. It evaluates the light-producing capacity, or luminous efficiency, of radiant energy.

(*c*) *Spectral Irradiance*, E_λ.—The density of radiant flux incident on an element of surface per unit wave length.

NOTE.—A source characterized by values of spectral irradiance constant throughout the spectrum is known as an "equal-energy source."

(*d*) *Tristimulus Designations*, X_o, Y_o, Z_o.—The color designations of the standard used in the spectrophotometric measurements of the test specimen.

(*e*) *Trichromatic Coefficients*, x, y, z.—Tristimulus designations expressed as fractions of their total, as follows:

$$x \equiv \frac{X}{X+Y+Z}$$

$$y \equiv \frac{Y}{X+Y+Z}$$

$$z \equiv \frac{Z}{X+Y+Z}$$

(*f*) *Spectral Transmission*, T_λ.—The ratio of transmitted energy to the spectrally homogeneous energy incident on any test specimen.

(*g*) *Luminous Transmission*, T_s.—The ratio of transmitted to incident light.

NOTE.—Luminous transmission is a function of the spectral distribution of the incident energy. It may be calculated from spectral transmission, T_λ, and the spectral luminosity, $\bar{y}_\lambda$, as follows:

$$T_s = \frac{\sum_0^\infty T_\lambda E_\lambda \bar{y}_\lambda \Delta\lambda}{\sum_0^\infty E_\lambda \bar{y}_\lambda \Delta\lambda}$$

(*h*) *Spectral Reflectance*.—The ratio of the reflected energy to the spectrally homogeneous energy incident on any test specimen.

NOTE.—Spectral reflectance depends upon the angular distribution of the incident energy.

(*i*) *Spectral Reflectivity*.—The spectral reflectance of a test specimen consisting of a layer of material of sufficient thickness that any increase would fail to change this ratio.

NOTE.—Spectral reflectivity depends upon the angular distribution of the incident energy.

(*j*) *Spectral Apparent Reflectance*.—The spectral reflectance which an ideal diffusing surface would need to possess in order to yield the same appearance as the test specimen under the same illuminating and viewing conditions.

NOTE.—Spectral apparent reflectance depends upon the angular distribution of the incident light and upon the direction of viewing.

(*k*) *Spectral Apparent Reflectivity*, R_λ.—The spectral reflectance which an ideal diffusing surface would need to possess in order to yield the same appearance as the test material under the same illuminating and viewing conditions.

NOTE.—Spectral apparent reflectivity depends upon the angular distribution of the incident light and upon the direction of viewing. It differs from spectral apparent reflectance only because it characterizes a material rather than a specimen. The ending "ivity" is customarily reserved for this meaning.

(*l*) *Luminous Reflectance*.—The ratio of the light reflected by any test specimen to that incident on it.

NOTE.—Luminous reflectance depends upon the spectral and angular distribution of the incident energy.

(*m*) *Luminous Reflectivity*.—The luminous reflectance of a test specimen consisting of a layer of material of sufficient thickness that any increase would fail to change this ratio.

NOTE.—Luminous reflectivity depends upon the spectral and angular distribution of the incident energy.

(*n*) *Luminous Apparent Reflectance*.—The luminous reflectance which a perfectly diffusing surface would need to possess in order to appear exactly as bright as the test specimen under the

same illuminating and viewing conditions.

NOTE.—Luminous apparent reflectance depends upon the spectral and angular distribution of the incident energy and upon the direction of viewing.

(*o*) *Luminous Apparent Reflectivity, R_s.*—The luminous reflectance which a perfectly diffusing surface would need to possess in order to appear exactly as bright as the test material under the same illuminating and viewing conditions.

NOTE.—Luminous apparent reflectivity depends upon the spectral and angular distribution of the incident energy and upon the direction of viewing. It may be calculated from spectral apparent reflectivity, R_λ, and the spectral luminosity, $\bar{y}_\lambda$, as follows:

$$R_s = \frac{\sum_0^\infty R_\lambda E_\lambda \bar{y}_\lambda \Delta\lambda}{\sum_0^\infty E_\lambda \bar{y}_\lambda \Delta\lambda}$$

(*p*) *Spectral Characteristics.*—The appropriate quantity as a function of wave length by means of which the test specimen is characterized. Spectral transmission as a function of wave length characterizes a light-transmitting specimen; spectral apparent reflectivity characterizes a light-reflecting material; and spectral apparent reflectance characterizes a light-reflecting object.

Apparatus

3. The apparatus shall consist of a spectrophotometer by means of which the spectral characteristics of the test specimen may, within the limits of the visible spectrum, be determined. Such apparatus may be of either the visual or the photoelectric type, but shall be suitable for determinations throughout the visible spectrum. The apparatus shall be so designed as to provide for the elimination of stray energy. This may be done by such means as the introduction of stray-energy filters or the use of two dispersing prisms.

Standards

4. (*a*) For transparent specimens, the incident light shall be used as a standard relative to which the transmitted light is evaluated.

(*b*) For reflecting specimens, either of the following shall be used as a standard:

(*1*) The ideal, completely reflecting, completely diffusing surface, or

(*2*) A layer (at least 0.5 mm. in thickness) of magnesium oxide, freshly prepared by collecting the smoke from burning magnesium ribbon or shavings on a suitable surface.

NOTE.—For further information regarding the preparation of fumed standards, see "Preparation and Colorimetric Properties of a Magnesium-Oxide Reflectance Standard," *Letter Circular LC-547*, Nat. Bureau Standards, March, 1939.

Illumination and Viewing

5. (*a*) The angular distribution of the light incident on the test specimen shall be specified, stating whether the illumination is diffuse, nearly diffuse, or nearly unidirectional. If nearly unidirectional, the direction and angular size of the source shall be specified. Unless there is a special reason for doing otherwise, light-transmitting specimens shall be illuminated only from directions near the perpendicular to the surface.

(*b*) If a test specimen be viewed by reflected light the angular distribution of the light incident on it shall be identical with that incident on any standard, such as a layer of magnesium oxide, with which it is compared.

(*c*) The direction and angular size of the viewing element shall be specified.

NOTE.—This method of measuring the spectral characteristics of a test specimen serves to specify those of its optical properties which are responsible for its color, subject, however, to the limitation that the color may depend upon the manner of illumination and viewing. For glossy samples in particular, it is important to specify the manner of illumination and viewing, and the most essential single characteristic is whether or not the specular component of reflected energy is included in the measurement.

Since glossy samples are customarily examined for color in such a way as to avoid specularly reflected light as much as possible, it is recommended that in these measurements also, the specular component be excluded by proper choice of illuminating and viewing conditions (such as 45-deg. illumination with perpendicular viewing, as recommended in 1931 by the International Commission on Illumination) or by application of a suitable correction. It should be noted that a light-reflecting specimen requires for specification of its appearance, in addition to the above, some measure of its gloss. This procedure is beyond the intended scope of this method and such measurement of gloss shall be made in accordance with the Tentative Method of Test for Specular Gloss of Paint Finishes (A.S.T.M. Designation: D 523) of the American Society for Testing Materials.[4]

Calibrations

6. (*a*) The calibration of the photometer scale, or other intensity-measuring unit, shall be carefully checked at reasonable time intervals in a manner to insure accuracy over the entire range.

(*b*) The accuracy of the wave-length calibration shall be adequately checked at reasonable time intervals.

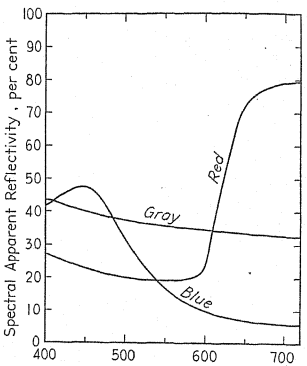
(*c*) The slit width shall be adjusted so that slit-width corrections are negligible; otherwise these corrections shall be applied.

Procedure

7. (*a*) The test specimen shall be handled carefully to avoid discoloration, and care shall be taken not to touch the area to be tested except for application of a suitable cleaning process. The condition of the test specimen before and after analysis shall be carefully noted and any change in the appearance of the surface of the tested area shall be recorded.

(*b*) Determination of spectral characteristics relative to the standard shall be made throughout such wave-length range within the visible spectrum and at such wave-length intervals that the curve drawn by interpolation and extrapolation would not be changed significantly by taking further readings.

NOTE.—Readings at 20 mμ intervals between 400 and 700 mμ are often sufficient; 10 mμ intervals between 380 and 770 mμ usually are sufficient. Typical curves of flat paints are shown in Fig. 1.



NOTE.—The red, gray, and blue paints were prepared by extending cadmium red, ivory black, and Prussian blue, respectively, with zinc white.

FIG. 1.—Typical Curves of Spectral Apparent Reflectivity.

(*c*) If spectral apparent reflectivity of a material is being determined, the test specimen shall consist of a layer of the material so thick that further increase in thickness will not cause a change in appearance (see Note 2, Section 1).

Rejection

9. If the test specimen undergoes a visible change during the analysis, the readings shall be rejected (see Section 7 (*a*)).

Report

10. The report of the spectral characteristics of a test specimen shall consist of the following:

(*a*) The spectral transmission, T_λ, of a light-transmitting object, or the spec-

[4] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

tral apparent reflectance of a light-reflecting object, or the spectral apparent reflectivity, R_λ, of a light-reflecting material.

NOTE.—If the test specimen is a translucent object which both transmits and reflects appreciable quantities of light, it may be necessary to report both its spectral transmission and its spectral apparent reflectance.

(*b*) The manner of illumination and manner of viewing, as specified in Section 5 (*a*) and (*c*).

General Plan of Color Designation

11. The color of a nonself-luminous specimen depends both upon the spectral composition of the illuminant and upon the spectral characteristics of the specimen itself. The method of designating the color of a material or object under a particular illuminant is to evaluate the light leaving it on each of three scales, an *X*-scale, a *Y*-scale, and a *Z*-scale. The color of a light-transmitting object for a particular illuminant should be designated by its luminous transmission, T_s, and trichromatic coefficients, x and y, for that illuminant. Similarly, the color of a reflecting material under a particular illuminant should be designated by its luminous apparent reflectivity, R_s, and its trichromatic coefficients, x and y, for that illuminant. Also, the color of a reflecting object under a particular illuminant should be designated by its luminous apparent reflectance and its trichromatic coefficients, x and y, for that illuminant. These quantities may be found from the values of X, Y, and Z for the test specimen and standard as described in Sections 12 and 13.

Standard Illuminants

12. Standard illuminant A shall be used if the color of the test specimen in incandescent lamplight is of interest, standard illuminant B for the color of the specimen in sunlight, and standard illuminant C for the color of the specimen in average daylight.[5] The relative spectral irradiance, E_λ, of these three

TABLE I.—RELATIVE SPECTRAL IRRADIANCE OF THE THREE STANDARD ILLUMINANTS, A, B, AND C.

Wave Length, mμ	E_A	E_B	E_C	Wave Length, mμ	E_A	E_B	E_C
380	9.79	22.40	33.00	580	114.44	101.00	97.80
385	10.90	26.85	39.92	585	118.08	100.07	95.43
390	12.09	31.30	47.40	590	121.73	99.20	93.20
395	13.36	36.18	55.17	595	125.39	98.44	91.22
400	14.71	41.30	63.30	600	129.04	98.00	89.70
405	16.15	46.62	71.81	605	132.70	98.08	88.83
410	17.68	52.10	80.60	610	136.34	98.50	88.40
415	19.29	57.70	89.53	615	139.99	99.06	88.19
420	21.00	63.20	98.10	620	143.62	99.70	88.10
425	22.79	68.37	105.80	625	147.23	100.36	88.06
430	24.67	73.10	112.40	630	150.83	101.00	88.00
435	26.64	77.31	117.75	635	154.42	101.56	87.86
440	28.70	80.80	121.50	640	157.98	102.20	87.80
445	30.85	83.44	123.45	645	161.51	103.05	87.99
450	33.09	85.40	124.00	650	165.03	103.90	88.20
455	35.41	86.88	123.60	655	168.51	104.59	88.20
460	37.82	88.30	123.10	660	171.96	105.00	87.90
465	40.30	90.08	123.30	665	175.38	105.08	87.22
470	42.87	92.00	123.80	670	178.77	104.90	86.30
475	45.52	93.75	124.09	675	182.12	104.55	85.30
480	48.25	95.20	123.81	680	185.43	103.90	84.00
485	51.04	96.23	122.92	685	188.70	102.84	82.21
490	53.91	96.50	120.70	690	191.93	101.60	80.20
495	56.85	95.71	116.90	695	195.12	100.38	78.24
500	59.86	94.20	112.10	700	198.26	99.10	76.30
505	62.93	92.37	106.98	705	201.36	97.70	74.36
510	66.06	90.70	102.30	710	204.41	96.20	72.40
515	69.25	89.65	98.81	715	207.41	94.60	70.40
520	72.50	89.50	96.90	720	210.36	92.90	68.30
525	75.79	90.43	96.78	725	213.26	91.10	66.30
530	79.13	92.20	98.00	730	216.12	89.40	64.40
535	82.52	94.46	99.94	735	218.92	88.00	62.80
540	85.95	96.90	102.10	740	221.66	86.90	61.50
545	89.41	99.16	103.95	745	224.36	85.90	60.20
550	92.91	101.00	105.20	750	227.00	85.20	59.20
555	96.44	102.20	105.67	755	229.58	84.80	58.50
560	100.00	102.80	105.30	760	232.11	84.70	58.10
565	103.58	102.92	104.11	765	234.59	84.90	58.00
570	107.18	102.60	102.30	770	237.01	85.40	58.20
575	110.80	101.90	100.15	775	239.37	86.10	58.50
580	114.44	101.00	97.80	780	241.67	87.00	59.10

standard illuminants throughout the visible spectrum is given in Table I, and the trichromatic coefficients, x, y, z, of

[5] For information regarding the procurement of lamps and liquid filters required to duplicate these standard illuminants, reference should be made to the following articles:

Proceedings, Eighth Session, Commission Internationale de l'Éclairage, Cambridge, England, September, 1931, pp. 19-29.

A. C. Hardy, "Handbook of Colorimetry," p. 16, Technology Press, Cambridge, Mass. (1936).

D. B. Judd, "The 1931 I.C.I. Standard Observer and Coordinate System for Colorimetry," *Journal*, Optical Soc. America, Vol. 23, p. 359 (1933).

R. Davis and K. S. Gibson, "Filters for the Reproduction of Sunlight and Daylight and the Determination of Color Temperature," *Miscellaneous Publication No. 114*, Nat. Bureau Standards (1931).

the standard illuminants are given in Table II.

TABLE II.—TRICHROMATIC COEFFICIENTS OF THE STANDARD ILLUMINANTS.

Standard Illuminant	Trichromatic Coefficients		
	x	y	z
A.....	0.4476	0.4075	0.1449
B.....	0.3484	0.3516	0.3000
C.....	0.3101	0.3163	0.3736

Procedure

13. (*a*) The spectral composition of the light leaving a nonself-luminous reflecting material is found by multiplying, for each wave length of the visible spectrum, the spectral irradiance, E_λ, of the illuminant by the spectral apparent reflectivity, R_λ, of the material; that is, the product, $R_\lambda E_\lambda$, is found throughout the visible spectrum (380 to 770 mμ). If magnesium oxide is used as the standard, the spectral apparent reflectivity of the test specimen should be expressed relative to that for magnesium oxide.

NOTE 1.—The evaluation of X, Y, and Z for a transparent object viewed by transmitted light, or for an opaque object viewed by reflected light, is found in a way strictly analogous to that for an opaque layer of material viewed by reflected light. The spectral transmission, T_λ, and spectral apparent reflectance of an object serve the same purpose as the spectral apparent reflectivity, R_λ, of a reflecting material.

(*b*) The light leaving the specimen of spectral composition, $R_\lambda E_\lambda$, is evaluated on the X-scale for each part of the spectrum by multiplying the corresponding $R_\lambda E_\lambda$ by the X-value for unit irradiance for that part of the spectrum. This light is also evaluated on the Y, and Z scales in the same way. Table III gives the X, Y, and Z values for unit irradiance throughout the visible spectrum for each 5-mμ interval. These values are identified by the symbols, $\bar{x}_\lambda$, $\bar{y}_\lambda$, $\bar{z}_\lambda$, respectively. From this step the products, $R_\lambda E_\lambda \bar{x}_\lambda$, $R_\lambda E_\lambda \bar{y}_\lambda$, and $R_\lambda E_\lambda \bar{z}_\lambda$ are found throughout the visible spectrum.

(*c*) The light leaving the specimen of spectral composition, $R_\lambda E_\lambda$, is evaluated on the X-scale for the whole spectrum by adding together the values of X found for the intervals of the spectrum separately. The light is also evaluated on the Y and Z scales in the same way. From this step are found the three numbers, X, Y, and Z:

$$X \equiv \sum_0^\infty R_\lambda E_\lambda \bar{x}_\lambda \Delta\lambda$$

$$Y \equiv \sum_0^\infty R_\lambda E_\lambda \bar{y}_\lambda \Delta\lambda$$

$$Z \equiv \sum_0^\infty R_\lambda E_\lambda \bar{z}_\lambda \Delta\lambda$$

NOTE 2.—For materials of usual spectral selectivity, an accurate evaluation of X, Y, and Z is obtainable by setting $\Delta\lambda = 10$ mμ; but for some materials it is found necessary to take $\Delta\lambda = 5$ mμ or even less. For interpolated values of $\bar{x}_\lambda$, $\bar{y}_\lambda$, and $\bar{z}_\lambda$ for each millimicron from 380 to 770 mμ, see A. C. Hardy, "Handbook of Colorimetry," Technology Press, Cambridge (1936). The selected-ordinate method of calculating X, Y, and Z, in many circumstances the most rapid, is also described there.[6,7]

(*d*) Calculate the luminous apparent reflectivity, R_s, and two of the three

[6] For further information regarding methods of carrying out these calculations, reference should be made to the following articles:

T. Smith, "Condensed Tables for Colour Computation," *Proceedings*, Physical Soc., London, Vol. 46, p. 372 (1934).

D. Nickerson, "Disk Colorimetry; Including a Comparison of Methods for Computing Tristimulus Values for Certain Disks," *Journal*, Optical Soc. America, Vol. 25, p. 253 (1935).

H. W. Swank and M. G. Mellon, "A Calculator for Obtaining Tristimulus Values from Spectrophotometric Data," *Journal*, Optical Soc. America, Vol. 27, p. 414 (1937).

F. W. Sears, "An Improved Calculator for Obtaining Tristimulus Values from Spectrophotometric Curves," *Journal*, Optical Soc. America, Vol. 29, p. 77 (1939).

J. A. Van den Akker, "A Mechanical Integrator for Evaluating the Integral of the Product of Two Functions and Its Application to the Computation of I.C.I. Color Specifications from Spectrophotometric Curves," *Journal*, Optical Soc. America, Vol. 29, p. 364 (1939).

[7] For attachments to the spectrophotometer to perform these calculations automatically, reference should be made to the following articles:

A. C. Hardy, "A New Recording Spectrophotometer," *Journal*, Optical Soc. America, Vol. 25, p. 305 (1935).

J. Razek, "Electrical Device for the Determination of Trilinear Coordinates for the Razek-Mulder Color Analyzer," *Journal*, Optical Soc. America, Vol. 24, p. 54 (1934).

trichromatic coefficients x and y, as follows:

$$R_s = \frac{Y}{Y_o}$$

$$x \equiv \frac{X}{X + Y + Z}$$

$$y \equiv \frac{Y}{X + Y + Z}$$

standard (see Table II) are found by calculating the following ratios:

$$\frac{X_o}{X_o + Y_o + Z_o}, \text{ and}$$

$$\frac{Y_o}{X_o + Y_o + Z_o}$$

Table III gives the trichromatic coeffi-

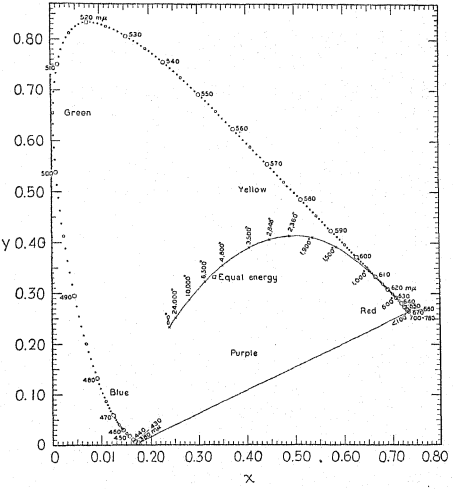


FIG. 2.—The (x, y)-Diagram Showing the Spectrum Colors.

It is customary to use a plot of y against x in rectangular coordinates (called a chromaticity diagram or mixture diagram) as an aid in determining the relation of the color of the specimen to known colors. For this purpose the colors of the spectrum and that of the standard are most frequently used. The trichromatic coefficients of the cients, x, y, z, of the spectrum colors, and the spectrum colors on the (x, y)-diagram are shown in Fig. 2.

NOTE 3.—If the specimen is a transparent object, the ratio, Y/Y_o, should be designated the luminous transmission; if it is a reflecting object consisting of a top translucent layer backed by a different material, the ratio Y/Y_o should be designated the luminous apparent reflectance.

Report

14. The report of the color designation of the test specimen shall consist of the following:

(*a*) The trichromatic coefficients, x and y.

specimen is a reflecting material, a reflecting object, or a light-transmitting object, respectively.

(*c*) The manner of illumination and manner of viewing as specified in Section 5 (*a*) and (*c*).

TABLE III.—THE 1931 I.C.I. STANDARD OBSERVER.

Trichromatic Coefficients			Wave Length, mμ	Tristimulus Specifications of the Equal-energy Spectrum			Trichromatic Coefficients			Wave Length, mμ	Tristimulus Specifications of the Equal-energy Spectrum		
x	y	z		$\bar{x}\lambda$	$\bar{y}\lambda$	$\bar{z}\lambda$	x	y	z		$\bar{x}\lambda$	$\bar{y}\lambda$	$\bar{z}\lambda$
0.1741	0.0050	0.8209	380	0.0014	0.0000	0.0065	0.5125	0.4866	0.0009	580	0.9163	0.8700	0.0017
0.1740	0.0050	0.8210	385	0.0022	0.0001	0.0105	0.5448	0.4544	0.0008	585	0.9786	0.8163	0.0014
0.1738	0.0049	0.8213	390	0.0042	0.0001	0.0201	0.5752	0.4242	0.0006	590	1.0263	0.7570	0.0011
0.1736	0.0049	0.8215	395	0.0076	0.0002	0.0362	0.6029	0.3965	0.0006	595	1.0567	0.6949	0.0010
0.1733	0.0048	0.8219	400	0.0143	0.0004	0.0679	0.6270	0.3725	0.0005	600	1.0622	0.6310	0.0008
0.1730	0.0048	0.8222	405	0.0232	0.0006	0.1102	0.6482	0.3514	0.0004	605	1.0456	0.5668	0.0006
0.1726	0.0048	0.8226	410	0.0435	0.0012	0.2074	0.6658	0.3340	0.0002	610	1.0026	0.5030	0.0003
0.1721	0.0048	0.8231	415	0.0776	0.0022	0.3713	0.6801	0.3197	0.0002	615	0.9384	0.4412	0.0002
0.1714	0.0051	0.8235	420	0.1344	0.0040	0.6456	0.6915	0.3083	0.0002	620	0.8544	0.3810	0.0002
0.1703	0.0058	0.8239	425	0.2148	0.0073	1.0391	0.7006	0.2993	0.0001	625	0.7514	0.3210	0.0001
0.1689	0.0069	0.8242	430	0.2839	0.0116	1.3856	0.7079	0.2920	0.0001	630	0.6424	0.2650	0.0000
0.1669	0.0086	0.8245	435	0.3285	0.0168	1.6230	0.7140	0.2859	0.0001	635	0.5419	0.2170	0.0000
0.1644	0.0109	0.8247	440	0.3483	0.0230	1.7471	0.7190	0.2809	0.0001	640	0.4479	0.1750	0.0000
0.1611	0.0138	0.8251	445	0.3481	0.0298	1.7826	0.7230	0.2770	0.0000	645	0.3608	0.1382	0.0000
0.1566	0.0177	0.8257	450	0.3362	0.0380	1.7721	0.7260	0.2740	0.0000	650	0.2835	0.1070	0.0000
0.1510	0.0227	0.8263	455	0.3187	0.0480	1.7441	0.7283	0.2717	0.0000	655	0.2187	0.0816	0.0000
0.1440	0.0297	0.8263	460	0.2908	0.0600	1.6692	0.7300	0.2700	0.0000	660	0.1649	0.0610	0.0000
0.1355	0.0399	0.8246	465	0.2511	0.0739	1.5281	0.7311	0.2689	0.0000	665	0.1212	0.0446	0.0000
0.1241	0.0578	0.8181	470	0.1954	0.0910	1.2876	0.7320	0.2680	0.0000	670	0.0874	0.0320	0.0000
0.1096	0.0868	0.8036	475	0.1421	0.1126	1.0419	0.7327	0.2673	0.0000	675	0.0636	0.0232	0.0000
0.0913	0.1327	0.7760	480	0.0956	0.1390	0.8130	0.7334	0.2666	0.0000	680	0.0468	0.0170	0.0000
0.0687	0.2007	0.7306	485	0.0580	0.1693	0.6162	0.7340	0.2660	0.0000	685	0.0329	0.0119	0.0000
0.0454	0.2950	0.6596	490	0.0320	0.2080	0.4652	0.7344	0.2656	0.0000	690	0.0227	0.0082	0.0000
0.0235	0.4127	0.5638	495	0.0147	0.2586	0.3533	0.7346	0.2654	0.0000	695	0.0158	0.0057	0.0000
0.0082	0.5384	0.4534	500	0.0049	0.3230	0.2720	0.7347	0.2653	0.0000	700	0.0114	0.0041	0.0000
0.0039	0.6548	0.3413	505	0.0024	0.4073	0.2123	0.7347	0.2653	0.0000	705	0.0081	0.0029	0.0000
0.0139	0.7502	0.2359	510	0.0093	0.5030	0.1582	0.7347	0.2653	0.0000	710	0.0058	0.0021	0.0000
0.0389	0.8120	0.1491	515	0.0291	0.6082	0.1117	0.7347	0.2653	0.0000	715	0.0041	0.0015	0.0000
0.0743	0.8338	0.0919	520	0.0633	0.7100	0.0782	0.7347	0.2653	0.0000	720	0.0029	0.0010	0.0000
0.1142	0.8262	0.0596	525	0.1096	0.7932	0.0573	0.7347	0.2653	0.0000	725	0.0020	0.0007	0.0000
0.1547	0.8059	0.0394	530	0.1655	0.8620	0.0422	0.7347	0.2653	0.0000	730	0.0014	0.0005	0.0000
0.1929	0.7816	0.0255	535	0.2257	0.9149	0.0298	0.7347	0.2653	0.0000	735	0.0010	0.0004	0.0000
0.2296	0.7543	0.0161	540	0.2904	0.9540	0.0203	0.7347	0.2653	0.0000	740	0.0007	0.0003	0.0000
0.2658	0.7243	0.0099	545	0.3597	0.9803	0.0134	0.7347	0.2653	0.0000	745	0.0005	0.0002	0.0000
0.3016	0.6923	0.0061	550	0.4334	0.9950	0.0087	0.7347	0.2653	0.0000	750	0.0003	0.0001	0.0000
0.3373	0.6589	0.0038	555	0.5121	1.0002	0.0057	0.7347	0.2653	0.0000	755	0.0002	0.0001	0.0000
0.3731	0.6245	0.0024	560	0.5945	0.9950	0.0039	0.7347	0.2653	0.0000	760	0.0002	0.0001	0.0000
0.4087	0.5896	0.0017	565	0.6784	0.9786	0.0027	0.7347	0.2653	0.0000	765	0.0001	0.0000	0.0000
0.4441	0.5547	0.0012	570	0.7621	0.9520	0.0021	0.7347	0.2653	0.0000	770	0.0001	0.0000	0.0000
0.4788	0.5202	0.0010	575	0.8425	0.9154	0.0018	0.7347	0.2653	0.0000	775	0.0000	0.0000	0.0000
0.5125	0.4866	0.0009	580	0.9163	0.8700	0.0017	0.7347	0.2653	0.0000	780	0.0000	0.0000	0.0000
Totals											21.3713	21.3714	21.3715

(*b*) The luminous apparent reflectivity, R_s, the luminous apparent reflectance, or the luminous transmission, T_s, depending upon whether the test

(*d*) The illuminant to which the color designation of the test specimen applies; that is, whether the standard illuminant, A, B, or C, or some other illuminant.

Standard Method of Test for

ABRASION RESISTANCE OF ORGANIC COATINGS WITH THE AIR BLAST ABRASION TESTER[1]

A.S.T.M. Designation: D 658 – 44

ADOPTED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 658; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test is primarily intended for determining the abrasion resistance of organic coatings when applied to flat sheet metal of uniform surface texture.

Nature of Test

2. In this method the abrasion resistance of organic coatings is determined by the amount of abrasive required to wear through a unit film thickness of the coating when the abrasive is allowed to impinge on the coating at a uniform rate, under the action of a controlled air blast.

Apparatus

3. The apparatus shall consist of the following:

(*a*) *Abrasion Tester.*—An abrasion tester such as is illustrated in Figs. 1 and 2.

(*b*) *Air-Control Assembly.*—An air-control assembly such as is illustrated in Fig. 3.

Standardization of Abrasion Tester

4. The abrasion tester, equipped with a standard glass nozzle, shall be assembled as illustrated in Fig. 1. The inner tube of the nozzle, as illustrated in Fig. 4, shall be connected by a flexible tubing to a suitable oil-free air supply, which shall be delivered from control apparatus (see Fig. 3) capable of maintaining the pressure within plus or minus 1.5 mm. of mercury of the specified test pressure. For purposes of standardization the test pressure shall be adjusted to 10 cm. of mercury and the abrasive outlet opened. The machine shall be run for 3 min. and shall deliver an average of 44 ± 1 g. of carborundum per minute. The diameter of the air tubing shall be sufficiently large to permit a

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, this method was published as tentative from 1942 to 1944.

flow of air of 2.5 cu. ft. per min. at the nozzle. When these conditions are realized, the machine may be considered standardized and suitable for abrasion resistance determinations.

NOTE.—For purposes of interlaboratory standardization, the machines may be further standardized by using as a basis for comparison a material mutually agreed upon by the purchaser and the seller. The abrasion resistance results thus obtained should agree within plus or minus 2.5 per cent.

Test Specimen

5. (*a*) The metal panels upon which the coating is applied shall be not more than 5 in. in length, 4 in. in width, and $\frac{1}{4}$ in. in thickness (Note). The surface preparation of the panels shall be agreed upon by the purchaser and the seller.

NOTE.—The method may also be adapted for the testing of larger specimens or coated parts.

(*b*) The organic coating whose abrasion resistance is to be determined shall be applied to the clean metal panels in such a manner that a coating of uniform thickness is obtained.[3] The time between application of the coating and the testing of the specimen, as well as the temperature and humidity environment used for maturing the coating during this period, shall be agreed upon by the purchaser and the seller.

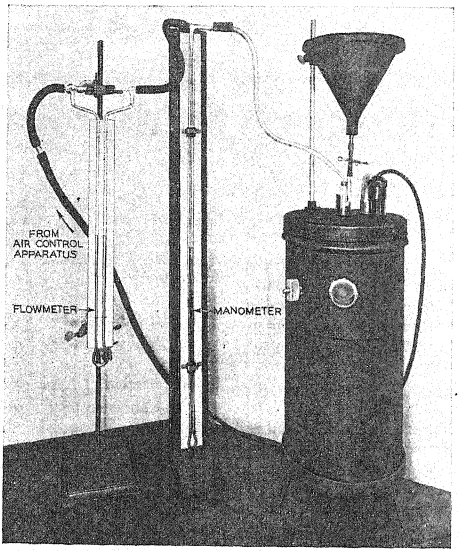


FIG 1.—Abrasion Test Apparatus (Closed).

[3] See papers by P. H. Walker and J. G. Thompson on "Some Physical Properties of Paints," *Proceedings*, Am. Soc. Testing Mats., Vol. 22, Part II, p. 464 (1922); and by H. G. Arlt, Bell Laboratories *Record*, Vol. XIV, No. 7, p. 216 (1936).

Conditioning and Number of Tests

6. At least three determinations of the abrasion resistance shall be taken under conditions of temperature and humidity mutually agreed upon by the purchaser and the seller. If it is desirable to check abrasion values at another time or place, the film thickness and the temperature and humidity conditions at the time of test shall be the same as those at which the original determinations were made. Unless otherwise specified, abrasion resistance determinations shall be made at 77 ± 2 F. and 50 ± 5 per cent relative humidity after equilibrating to these conditions.

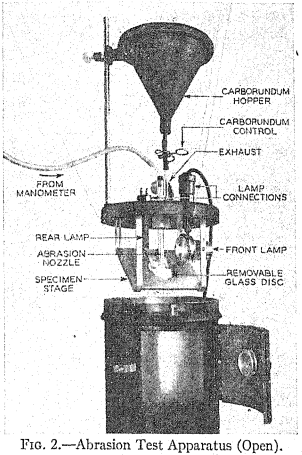


FIG. 2.—Abrasion Test Apparatus (Open).

Procedure

7. The abrasive holder shall be filled with carborundum[4] passing a No. 170 (88-micron) sieve and retained on a No. 200 (74-micron) sieve[5] (Note), or with equivalent abrasive, weighed on a balance sensitive to within 0.5 g., and then placed in position on the abrasion tester. The test specimen shall be placed on the specimen stage, which shall be mounted at an angle of 45 deg. to the horizontal, and the nozzle shall be lowered to touch the surface of the specimen. The air shall be turned on and adjusted to the specified pressure (usually 10 cm. of mercury) by manipulating the control valve. The stream of abrasive shall then be released, by removing the pinchcock attached to the

[4] Suitable abrasive may be obtained from the Carborundum Co., Niagara Falls, N. Y., as silicon carbide grain 180 special. It should be sieved before use and only the fraction employed that passes the No. 170 (88-micron) sieve and is retained on the No. 200 (74-micron) sieve.

[5] Detailed requirements for these sieves are given in the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

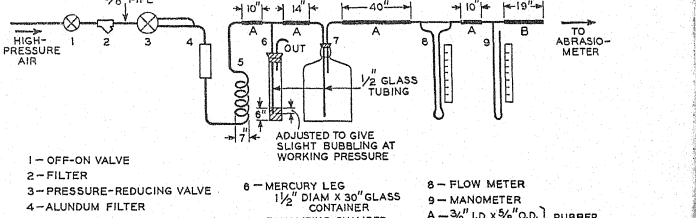


FIG. 3.—Air-Control Assembly for Abrasion Test.

holder, and allowed to flow until the coating is worn through to the base metal. The end point shall be considered as having been reached when a small spot approximately 2 mm. in diameter is worn through. This spot shall be located in the center of the wear pattern. When this point is reached the abrasive stream shall be immediately shut off and the weight of abrasive used shall be determined. The thickness of the film around the wear pattern shall then be determined by any method accurate to plus or minus 0.0001 in.

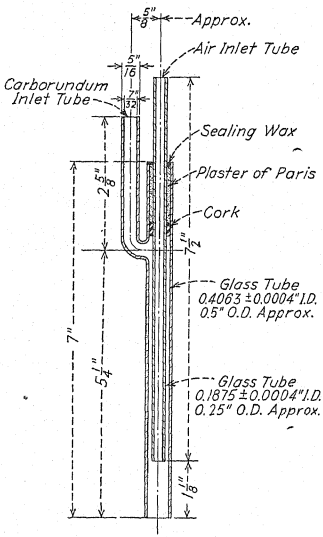


FIG. 4.—Glass Nozzle.

NOTE.—The used carborundum from a previous test may be reused provided it is sieved through a No. 100 (149-micron) sieve[5] before it is returned to the holder in order to eliminate all of the large particles of organic coating that chipped off during the abrasion test. The carborundum shall be replaced after it has been used five times.

Calculation

8. The abrasion coefficient for the material shall be calculated from the following formula in which it is assumed that the abrasion resistance is proportional to film thickness:

$$\text{Abrasion coefficient} = \frac{W_1 - W_2}{T}$$

where:

W_1 = weight in grams of abrasive and holder before test,

W_2 = weight in grams of abrasive and holder after test, and

T = thickness of coating in mils (0.001 in.).

Report

9. The average of the results from the determinations made shall be reported as the abrasion coefficient of the material.

Standard Method of

EVALUATING DEGREE OF RESISTANCE TO RUSTING OBTAINED WITH PAINT ON IRON OR STEEL SURFACES[1]

A.S.T.M. Designation: D 610 – 43

ADOPTED, 1943.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 610; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. The photographic reference standards[3] included in this method are representative of degrees of resistance to rusting obtained from paint on iron or steel surfaces. They are to be used for comparative purposes and are not intended to have a direct relationship to a decision regarding painting requirements.

Types of Rusting

2. Two types of rusting are represented:

Type 1.—Rusting not accompanied by blistering, and evidenced by visible rust.

Type 2.—Rusting accompanied by blistering, and not initially evidenced by visible rust.

Use of Photographic Reference Standards

3. The use of the photographic reference standards shown in Fig. 1 requires the following precautions:

(*a*) Some finishes are stained by rust. This staining must not be confused with the actual rusting involved.

(*b*) Accumulated dirt or other material may make accurate determination of the degree of rusting difficult.

(*c*) Certain types of deposited dirt that contain iron or iron compounds may cause surface discoloration that should not be mistaken for corrosion.

(*d*) It must be realized that failure may vary over a given area and discretion must therefore be used in applying these reference standards.

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[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

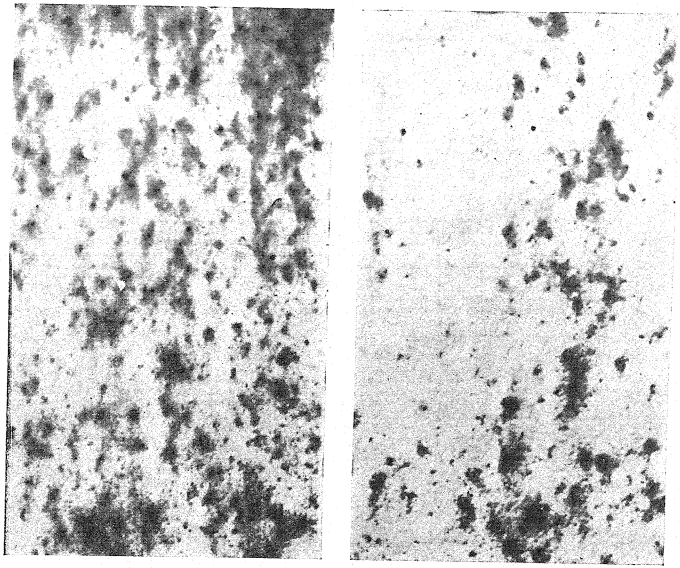
[2] Prior to adoption as standard, this method was published as tentative from 1941 to 1943.

[3] Selections of photographic reference standards were made to agree within practical limits with the degree of resistance to rusting indicated for the corresponding reference standard numbers in the Swedish Rusting Standards. The committee suggests that degrees below No. 4 are of no practical importance.

This Method, including the Photographic Reference Standards, is available in the form of a 3 by 5-in. collapsible folder furnished with a stiff cardboard case at one dollar each from the American Society for Testing Materials, 260 S. Broad St., Philadelphia 2, Pa.

(*e*) In evaluating surfaces, consideration shall be given to the color of the finish coating, since failures will be more apparent on a finish that shows color contrast with rust, such as used in these reference standards, than on a similar color, such as an iron oxide finish.

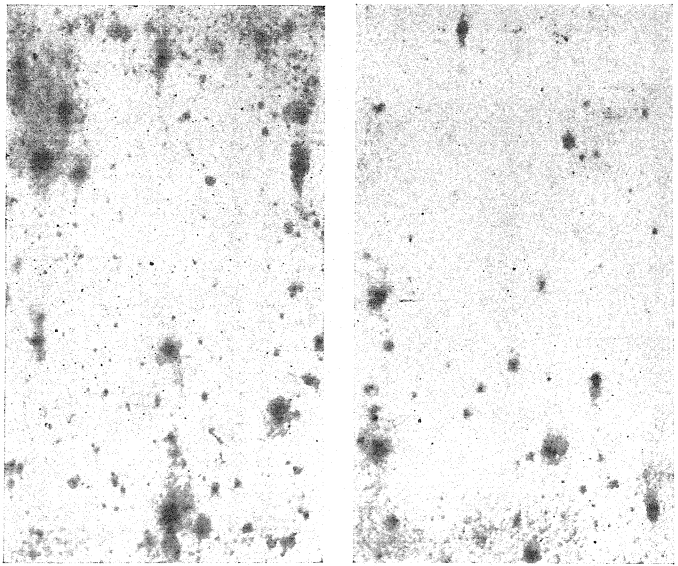
FIG. 1.—Photographic Reference Standards for Evaluating Degree of Resistance to Rusting Obtained with Paint on Iron or Steel Surfaces.



No. 4—1 No. 6—1

TYPE 1.—Rusting Not Accompanied by Blistering.

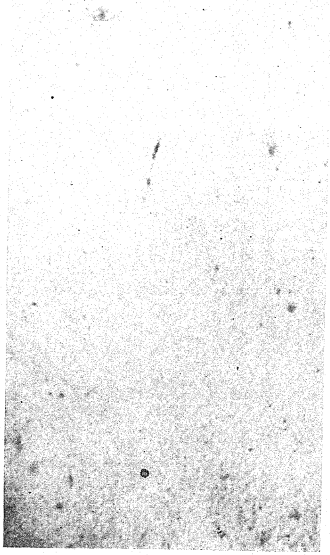
FIG. 1.—Photographic Reference Standards for Evaluating Degree of Resistance to Rusting Obtained with Paint on Iron or Steel Surfaces. (*Continued*)



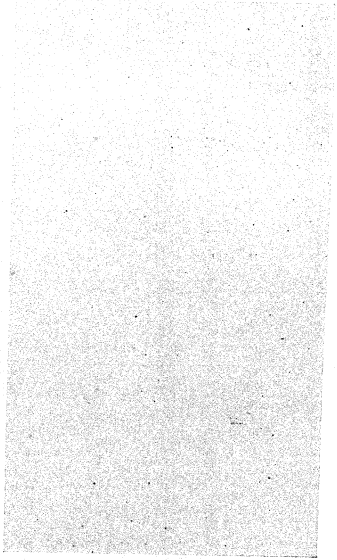
No. 7—1 No. 8—1

TYPE 1.—Rusting Not Accompanied by Blistering (*Continued*).

FIG. 1.—Photographic Reference Standards for Evaluating Degree of Resistance to Rusting Obtained with Paint on Iron or Steel Surfaces. (*Continued*)



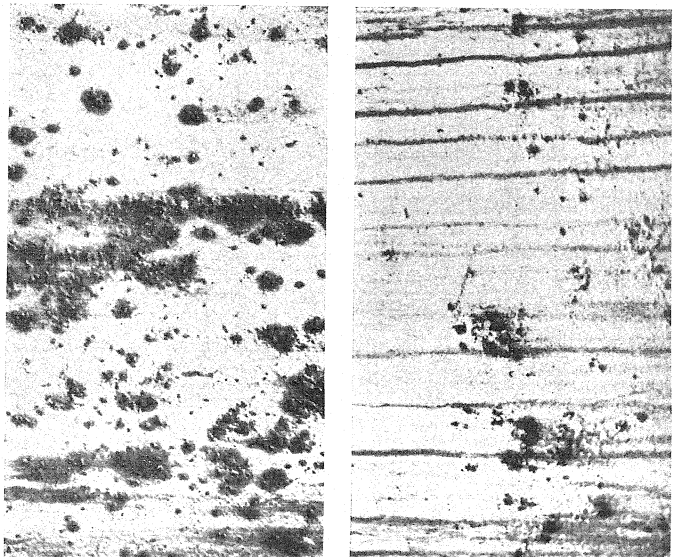
No. 9—1



No. 10—1

TYPE 1.—Rusting Not Accompanied by Blistering (*Continued*).

FIG. 1.—Photographic Reference Standards for Evaluating Degree of Resistance to Rusting Obtained with Paint on Iron or Steel Surfaces. (*Continued*)



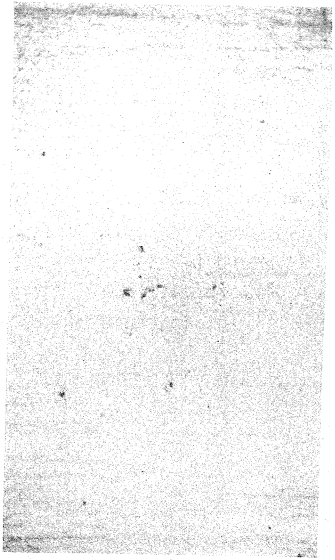
No. 4—2 No. 6—2

TYPE 2.—Rusting Accompanied by Blistering.

FIG. 1.—Photographic Reference Standards for Evaluating Degree of Resistance to Rusting Obtained with Paint on Iron or Steel Surfaces. (*Continued*)



No. 7—2



No. 8—2

TYPE 2.—Rusting Accompanied by Blistering (*Continued*).

Standard Method of

EVALUATING DEGREE OF RESISTANCE TO CHALKING OF EXTERIOR PAINTS OF THE LINSEED-OIL TYPE[1]

A.S.T.M. Designation: D 659 – 44

ADOPTED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 659; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. The photographic reference standards included in this method are representative of degrees of resistance to chalking of linseed-oil-type exterior paint films on wood surfaces. These standards are primarily intended for comparative evaluation.

Definition

2. Chalking is that phenomenon manifested in paint films by the presence of loose removable powder, evolved from the film itself, at or just beneath the surface. Chalking may be detected by rubbing the film with the fingertip.

Types of Chalking

3. Only one type of chalking is recognized, as defined in Section 2.

Procedure

4. Wrap a piece of black velvet, flannel, or felt around the tip of the index finger and apply, with medium pressure, to the film under observation by making a stroke across the surface at least 4 in. in length. Shift the index finger to a position 1 in. to the side of the chalk-retaining spot and make another reading. Compare the spots thus obtained with the photographic standard.

Use of Photographic Reference Standards

5. The use of the photographic reference standards[3] shown in Fig. 1 requires the following precautions:

(*a*) It must be realized that the degree of failure will vary over any given area. Therefore, an average portion of the film should be used for comparison.

(*b*) It is very difficult to make readings on a windy day and making readings at such a time should be avoided. It should also be noted that rain or snow will remove chalk, so that readings should be made after a period of clear weather and when the surface is dry.

(*c*) It should be remembered that

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

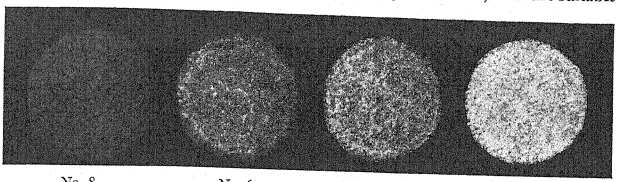
[2] Prior to adoption as standard, this method was published as tentative from 1942 to 1944.

[3] Copies of the Exposure Standards Manual prepared by the Federation of Paint and Varnish Production Clubs, giving actual photographs of various types of failures of exterior paints, may be obtained from the Secretary of the Federation, 704 Weightman Building, 1524 Chestnut St., Philadelphia 2, Pa.

chalking and erosion[4] are closely related and that erosion is a result of chalking failure. However, the rate of chalking, as measured by this method, and the rate of erosion may not be comparable, because some pigment combinations tend to retain chalk on the surface while other pigment combinations exert a self-cleaning action by natural means.

(*d*) While this method appears to be open to many variables, experience has shown that the results obtained may be readily reproduced. Several instruments have been designed to determine degrees of chalking, using this same principle but with controlled pressure. While they have merit, most are suitable only for laboratory examinations and require too much time for the evaluation of large numbers of panels.

(*e*) For convenience in recording the data obtained, the record sheet approved by the Federation of Paint and Varnish Production Clubs is recommended.[5]



No. 8 No. 6 No. 4 No. 2

FIG. 1.—Degrees of Chalking.

[4] For evaluation of erosion, see the Standard Method of Evaluating Degree of Resistance to Erosion of Exterior Paints of the Linseed-Oil Type (A.S.T.M. Designation: D 662), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

[5] These record sheets may be obtained from the Palmerton Printing Co., Palmerton, Pa., or from the American Society for Testing Materials, 260 S. Broad St., Philadelphia 2, Pa.

Standard Method of

EVALUATING DEGREE OF RESISTANCE TO CHECKING OF EXTERIOR PAINTS OF THE LINSEED-OIL TYPE[1]

A.S.T.M. Designation: D 660 - 44

ADOPTED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 660; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. The photographic reference standards included in this method are representative of degrees of resistance to checking of linseed-oil-type exterior paint films on wood surfaces. These standards are primarily intended for comparative evaluation.

Definition

2. Checking is that phenomenon manifested in paint films by slight breaks in the film that do not penetrate to the underlying surface. The break should be called a crack[3] if the underlying surface is visible. Where precision is necessary in evaluating a paint film, checking may be described as visible (as seen with the naked eye) or as microscopic (as observed under a magnification of 10 diameters).

Types of Checking

3. Three types of checking are recognized:

Irregular Pattern Type.—Checking in which the breaks develop in the surface of the film in no definite pattern.

Line Type.—Checking in which the breaks in the surface of the film are generally arranged in parallel lines, usually either horizontally or vertically, over the surface of the film. These breaks often follow the line of brush marks.

Crowfoot Type.—Checking in which the breaks in the surface of the film form in a definite three-prong pattern with the breaks running from a center and forming an angle of about 120 deg. between the prongs.

Use of Photographic Reference Standards

4. The use of the photographic reference standards[4] shown in Fig. 1 requires the following precautions:

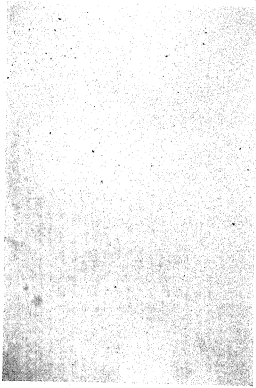
(*a*) The accompanying photographic

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

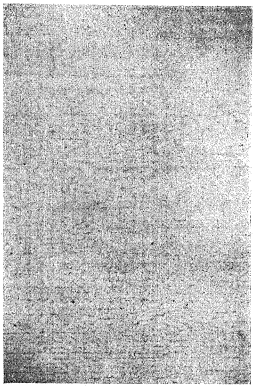
[2] Prior to adoption as standard, this method was published as tentative from 1942 to 1944.

[3] For evaluation of cracking, see the Standard Method of Evaluating Degree of Resistance to Cracking of Exterior Paints of the Linseed-Oil Type (A.S.T.M. Designation: D 661), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

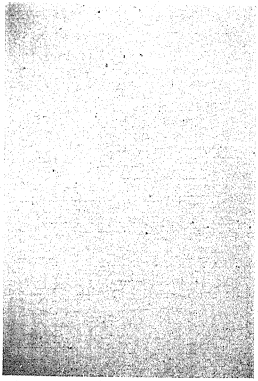
[4] Copies of the Exposure Standards Manual prepared by the Federation of Paint and Varnish Production Clubs, giving actual photographs of various types of failures of exterior paints, may be obtained from the Secretary of the Federation, 704 Weightman Building, 1524 Chestnut St., Philadelphia 2, Pa.



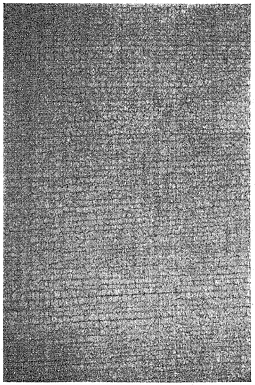
No. 8



No. 6



No. 4



No. 2

FIG. 1.—Degrees of Checking.

reference standards show line-type checking only. Crowfoot and irregular-type checking may also be interpreted from the photographs.

(*b*) Care must be taken not to confuse various types of failure that may be present on the same surface.

(*c*) It must be realized that the degree of failure will vary over any given area. Therefore, an average portion of the film should be used for comparison.

(*d*) Linseed-oil paint films often collect excessive quantities of dirt, which may mask the type and degree of failure. If necessary, dirt should be removed by careful and gentle brushing with a moderately soft brush.

(*e*) The use of a microscope is recommended to detect and evaluate incipient checking.

(*f*) The No. 8 standard must be examined closely under adequate lighting conditions to distinguish the failure present. For the sake of clarity, a 10-diameter magnification of No. 8 checking is shown in Fig. 2, as well as the unmagnified view shown in Fig. 1.



FIG. 2.—No. 8 Checking Magnified 10 Diameters.

(*g*) For convenience in recording the data obtained, the record sheet approved by the Federation of Paint and Varnish Production Clubs is recommended.[5]

[5] These record sheets may be obtained from the Palmerton Printing Co., Palmerton, Pa., or from the American Society for Testing Materials, 260 S. Broad St., Philadelphia 2, Pa.

Standard Method of

EVALUATING DEGREE OF RESISTANCE TO CRACKING OF EXTERIOR PAINTS OF THE LINSEED-OIL TYPE[1]

A.S.T.M. Designation: D 661 – 44

ADOPTED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 661; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. The photographic reference standards included in this method are representative of degrees of resistance to cracking of linseed-oil-type exterior paint films on wood surfaces. These standards are primarily intended for comparative evaluation.

Definition

2. Cracking is that phenomenon manifested in paint films by a break extending through to the surface painted. Where this is difficult to determine, the break should be called a crack only if the underlying surface is visible. The use of a magnification of 10 diameters is recommended in cases where it is difficult to differentiate between cracking and checking.[3]

Types of Cracking

3. Three types of cracking are recognized:

Irregular Pattern Type.—Cracking in which the breaks in the film are in no definite pattern.

Line Type.—Cracking in which the breaks in the film are generally arranged in parallel lines, usually either horizontally or vertically, over the surface of the film. These breaks often follow the line of brush marks.

Sigmoid Type.—Cracking in which the breaks in the film form a pattern consisting of curves meeting and intersecting, usually on a relatively large scale.

Use of Photographic Reference Standards

4. The use of the photographic reference standards[4] shown in Fig. 1 requires the following precautions:

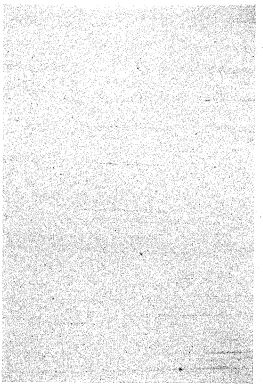
(*a*) The accompanying photographic

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

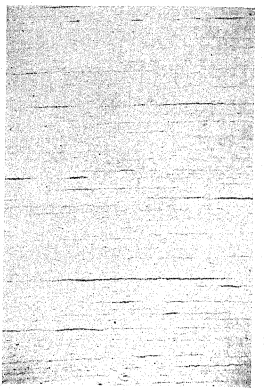
[2] Prior to adoption as standard, this method was published as tentative from 1942 to 1944.

[3] For evaluation of checking, see the Standard Method of Evaluating Degree of Resistance to Checking of Exterior Paints of the Linseed-Oil Type (A.S.T.M. Designation: D 660), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

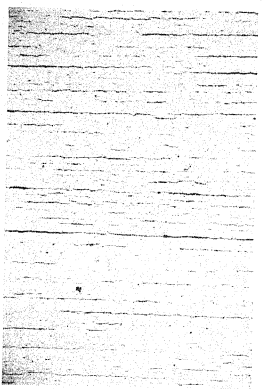
[4] Copies of the Exposure Standards Manual prepared by the Federation of Paint and Varnish Production Clubs, giving actual photographs of various types of failures of exterior paints, may be obtained from the Secretary of the Federation, 704 Weightman Building, 1524 Chestnut St., Philadelphia 2, Pa.



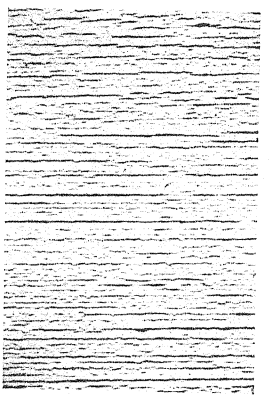
No. 8



No. 6



No. 4



No. 2

FIG. 1.—Degrees of Cracking.

reference standards show line-type cracking only. Irregular and sigmoid-type cracking may also be interpreted from these photographs.

(*b*) Care must be taken not to confuse various types of failure that may be present on the same surface. This is particularly true in observing cracking and checking. Cracking may very often be an advanced stage of checking and is very often in evidence along with checking and other failures.

(*c*) It must be realized that the degree of failure will vary over any given area. Therefore, an average portion of the film should be used for comparison.

(*d*) Linseed-oil paint films often collect excessive quantities of dirt, which may mask the type and degree of failure. If necessary, dirt should be removed by careful and gentle brushing with a moderately soft brush.

(*e*) In examining for cracking failure, the possibility of wood failure should be recognized. This takes the form of a cracking or splitting of the wood itself with a resultant rupture of the paint film. Also, some panels will develop "resin spewing" which will cause early failure by cracking. These points should be taken into consideration in any evaluations.

(*f*) For convenience in recording the data obtained, the record sheet approved by the Federation of Paint and Varnish Production Clubs is recommended.[5]

[5] These record sheets may be obtained from the Palmerton Printing Co., Palmerton, Pa., or from the American Society for Testing Materials, 260 S. Broad St., Philadelphia 2, Pa.

Standard Method of

EVALUATING DEGREE OF RESISTANCE TO EROSION OF EXTERIOR PAINTS OF THE LINSEED-OIL TYPE[1]

A.S.T.M. Designation: D 662 - 44

ADOPTED, 1944.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 662; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. The photographic reference standards included in this method are representative of degrees of resistance to erosion of linseed-oil-type exterior paint films on wood surfaces. These standards are primarily intended for comparative evaluation.

Definition

2. Erosion is that phenomenon manifested in paint films by the wearing away of the finish to expose the substrate. The degree of failure is dependent on the amount of substrate visible. Erosion occurs as the result of chalking.[3]

Types of Erosion

3. Only one type of erosion is recognized, as defined in Section 2.

Use of Photographic Reference Standards

4. The use of the photographic reference standards[4] shown in Fig. 1 requires the following precautions:

(*a*) Care must be taken not to confuse various types of failure that may be present on the same surface.

(*b*) It must be realized that the degree of failure will vary over any given area. Therefore, an average portion of the film should be used for comparison.

(*c*) The photographic standards used represent various degrees of erosion of a white brushing type paint over a dark primer. This system was necessary to provide sufficient contrast for photographic purposes. The erosion of a film to its normal substrate is, however, readily visible to the naked eye so it may easily be compared to the standards and given a numerical rating.

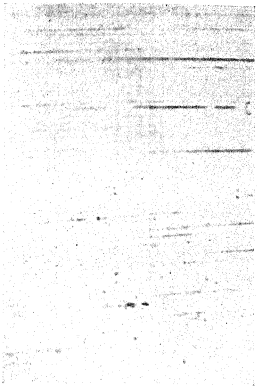
(*d*) In doubtful cases, erosion is some-

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, this method was published as tentative from 1942 to 1944.

[3] For evaluation of chalking, see the Standard Method of Evaluating Degree of Resistance to Chalking of Exterior Paints of the Linseed-Oil Type (A.S.T.M. Designation: D 659), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

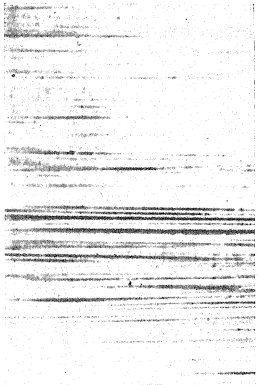
[4] Copies of the Exposure Standards Manual prepared by the Federation of Paint and Varnish Production Clubs, giving actual photographs of various types of failures of exterior paints, may be obtained from the Secretary of the Federation, 704 Weightman Building, 1524 Chestnut St., Philadelphia 2, Pa.



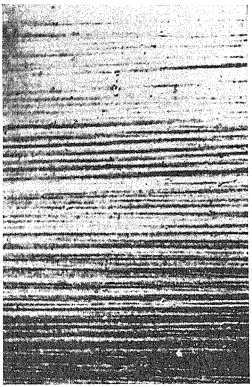
No. 8



No. 6



No. 4



No. 2

FIG. 1.--Degrees of Erosion.

times more visible in a damp film than in a dry film. Also, with severe erosion, it is often easier to rate the degree of erosion in a damp film than in a dry film.

(*e*) While erosion of a sprayed film is more regular in its wearing away, a numerical rating can be given to it by interpreting the amount of erosion in terms of these standards.

(*f*) For convenience in recording the data obtained, the record sheet approved by the Federation of Paint and Varnish Production Clubs is recommended.[5]

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[5] These record sheets may be obtained from the Palmerton Printing Co., Palmerton, Pa.; or from the American Society for Testing Materials, 260 S. Broad St., Philadelphia 2, Pa.

Standard Specifications for
PURE LINSEED OIL PUTTY FOR GLAZING[1]

A.S.T.M. Designation: D 317 – 33

ADOPTED, 1933.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation D 317; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. (*a*) These specifications cover two classes of putty for glazing and similar purposes, as follows:

Class A.—Whiting putty.

Class B.—White lead whiting putty.

(*b*) The putty shall be furnished in natural color, or tinted or colored, as specified by the purchaser.

Manufacture

2. The putty shall be made by thoroughly grinding or "chasing" the specified dry pigment with pure linseed oil to a dough-like paste of proper consistency.

Properties of Ingredients

3. (*a*) *Dry Pigment.*—The dry pigment shall conform to the following requirements:

	Whiting Putty	White Lead Whiting Putty
Calcium carbonate, min., per cent	95	..
White lead, min., per cent	..	10
Total calcium carbonate and white lead, min., per cent	..	95
Tinting pigment plus impurities, max., per cent	5	5

(*b*) *Whiting.*—The whiting used in both classes of putty shall be free from grit, shall be practically neutral, and shall have a structure and physical characteristics adapted to the manufacture of putty having the required physical properties.

(*c*) *White Lead.*—The white lead shall be pure basic lead carbonate.

(*d*) *Tinting Pigment.*—Tinting or coloring pigments, if used, shall be pure and shall be used in the minimum quantity to secure the specified tint or shade.

(*e*) *Linseed Oil.*—The liquid shall consist entirely of pure linseed oil.

Properties of Finished Putty

4. (*a*) The finished putty shall conform to the requirements prescribed in Table I.

(*b*) The finished putty, after working in the hands, shall have good plastic quality without sticking to the hands. It shall work readily and smoothly under a knife without crumbling or

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to adoption as standard, these specifications were published as tentative from 1930 to 1933, being revised in 1932 and 1933. Editorially revised and rearranged in 1939.

Special Marking

7. In addition to the special markings prescribed in Table I, each thermometer shall be marked with a serial number and the manufacturer's name or trade mark etched on the stem.

Scale Error

8. The error at any point of the scale up to the temperature limit prescribed in Table I when the thermometer is standardized in accordance with Section 9 shall not exceed the values prescribed in Table I.

Standardization

9. The thermometer shall be standardized immersed in the testing bath to the top of the mercury column unless other conditions of immersion are prescribed in Table I, and at the temperatures prescribed in Table I. (See Explanatory Notes)

Test for Permanency of Range

10. The test for permanency of range shall be made at the temperature prescribed in Table I and under the immersion conditions specified for the thermometer. The accuracy after this test shall be within the limit specified.

Case

11. The thermometer shall be supplied in a suitable case on which shall appear the marking prescribed in Table I.

EXPLANATORY NOTE ON PERIODIC CHECKING ON CHANGES OF BULB VOLUME OF GLASS THERMOMETERS

A mercury in glass thermometer, if properly made and used, is an extremely reliable instrument but small changes in volume of the thermometer bulb may occur, even though the thermometer may have been carefully annealed or aged.

Usually the bulb contracts slowly and as a result the thermometer may read higher after it has been placed in service than it did originally. These changes are more appreciable for short-range thermometers graduated in fractional degrees, and the users of such thermometers should check such instruments from time to time. When first received the thermometers should be checked every week or so, later these time intervals may be lengthened if the changes are not of consequence.

The ice-point method (reading in melting ice at 0 C. or 32 F.) has been found to be the most convenient and at the same time the most accurate method for checking changes in bulb volume. Other temperatures, such as the boiling point of water and of other pure substances, have been proposed for "fixed points," but have not been found entirely satisfactory owing to the difficulties of accurately obtaining these fixed temperatures.

Short-range thermometers graduated in fractional degrees may or may not be provided with an ice point reading if this temperature is not included in the given range. This extra point can be incorporated in the thermometer, but may be omitted for the sake of economy. In the case of distillation thermometers intended to be inserted in the neck of a flask, the ice-point graduation may be undesirable since the contraction chamber between the ice point and the first graduation may contain enough mercury to cause uncertain and variable readings. For this reason the ice-point graduation, unless it is an integral part of the range, is generally omitted from the specifications for distillation thermometers.

In the absence of the ice point other means must be provided for detecting changes in the readings, especially if the thermometer is fractionally graduated. The method most commonly used involves the comparison at one or more temperatures in a well-stirred, properly constructed liquid bath of the thermometer to be checked against another standardized thermometer of similar specifications to the test thermometer but provided with an ice point.

Where several thermometers make up a series or set for some specific method and these thermometers do not have ice points, a similar set may be provided with ice points, and these should be kept for use as reference standards for checking routine test thermometers.

Detailed instructions for making such comparisons and specifications for suitable testing apparatus, as well as other data on this subject, will be found in the paper by E. F. Mueller and R. M. Wilhelm, "Methods of Testing Thermometers," *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part I, p. 493 (1938).

EXPLANATORY NOTE ON ICE POINT DETERMINATIONS OF GLASS THERMOMETERS

Two methods have been in use for determining the ice point on thermometers graduated fractionally and with an open scale, such as those of the Kinematic type. One method requires that the thermometer remain at approximately room temperature for 72 hr. before the ice point is taken. The other stipulates that the ice point reading be taken immediately or within a definitely stated short period after heating to the test temperature. Changes in the correction of the thermometer may be observed by determining the changes in the ice point by either method.

Since it may not be practical to wait until the thermometer has rested for three or more days, the method as outlined below is suggested. These instructions are given in the form of a note which should appear in the table of corrections for the thermometer.

NOTE.—The tabulated corrections apply for the condition of immersion indicated provided the ice point reading taken after heating to.....for not less than three minutes is.....

If the ice point reading (taken in not less than 2 min. and not more than 5 min. after removal of the thermometer from the heated bath) is found to be higher (or lower) than stated, all other readings will be higher (or lower) to the same extent.

TABLE I.—REQUIREMENTS FOR A.S.T.M. THERMOMETERS.

Name	A.S.T.M. Partial Immersion[a]		A.S.T.M. Partial Immersion[b]	
A.S.T.M. Designation	E 1 (1C - 39)	E 1 (1F - 39)	E 1 (2C - 39)	E 1 (2F - 39)
Liquid	mercury	mercury	mercury	mercury
Filling above Liquid	nitrogen gas	nitrogen gas	nitrogen gas	nitrogen gas
Temperature Range	−20 to +150 C.	0 to +300 F.	−5 to +300 C.	+20 to +580 F.
Subdivisions	1 C.	2 F.	1 C.	2 F.
Total Length	303 to 307 mm. (11.93 to 12.09 in.)		379 to 383 mm. (14.92 to 15.08 in.)	
Stem Diameter	6.0 to 7.0 mm. (0.236 to 0.276 in.)		6.0 to 7.0 mm. (0.236 to 0.276 in.)	
Bulb Diameter	5.0 to 6.0 mm. (0.197 to 0.236 in.)		5.0 to 6.0 mm. (0.197 to 0.236 in.)	
Bulb Length	19 to 25 mm. (0.75 to 0.98 in.)		10 to 15 mm. (0.39 to 0.59 in.)	
Bottom of Bulb to Graduation Line at Distance	−18 C. 90 to 100 mm.	0 F. (3.54 to 3.94 in.)	0 C. 100 to 110 mm.	32 F. (3.94 to 4.33 in.)
Top of Thermometer to Graduation Line at Distance	150 C. 20 to 35 mm.	300 F. (0.79 to 1.38 in.)	300 C. 25 to 50 mm.	572 F. (0.98 to 1.97 in.)
Top Finish	glass ring	glass ring	glass ring	glass ring
Longer Graduation Lines at Each	5 C.	10 F.	5 C.	10 F.
Graduations Numbered at Each Multiple of	10 C.	20 F.	10 C.	20 F.
Immersion	76 mm.[c]	3 in.[c]	76 mm.[c]	3 in.[c]
Special Marking on Thermometer (See Section 7)	ASTM 76-mm. Imm.	ASTM 3-in. Imm.	ASTM 76-mm. Imm.	ASTM 3-in. Imm.
Scale Error at any point when standardized shall not exceed	0.5 C.	1 F.	1 C.	2 F.
Standardization	[d]	[d]	[e]	[e]
Marking on Case	A.S.T.M. −20 to +150 C., 76-mm. immersion	A.S.T.M. 0 to +300 F., 3-in. immersion	A.S.T.M. −5 to +300 C., 76-mm. immersion	A.S.T.M. 20 to 580 F., 3-in. immersion

[a] These requirements were previously specified in A.S.T.M. Standard D 182.
[b] These requirements were previously specified in A.S.T.M. Standard D 183.
[c] A line around the stem 76 mm. or 3 in. above the bottom of the bulb shall be etched on the thermometer.
[d] The thermometer shall be standardized for 76-mm. or 3-in. immersion and for the following temperatures of the emergent mercury column. These stem temperatures have been chosen as corresponding, on the average, to those likely to occur in the use of the thermometer.

Thermometer Reading	Average Temperature of Emergent Mercury Column	Thermometer Reading	Average Temperature of Emergent Mercury Column
50 C	35 C.	122 F	94 F.
100 C	48 C.	212 F	118 F.
150 C	55 C.	302 F	131 F.

[e] The thermometer shall be standardized for 76-mm. or 3-in. immersion and for the following temperatures of the emergent mercury column. These stem temperatures have been chosen as corresponding, on the average, to those likely to occur in the use of the thermometer.

Thermometer Reading	Average Temperature of Emergent Mercury Column	Thermometer Reading	Average Temperature of Emergent Mercury Column
50 C	35 C.	122 F	94 F.
100 C	49 C.	212 F	120 F.
150 C	61 C.	302 F	142 F.
200 C	70 C.	392 F	158 F.
250 C	76 C.	482 F	169 F.
300 C	80 C.	572 F	176 F.

TABLE I.—REQUIREMENTS FOR A.S.T.M. THERMOMETERS (*Continued*).

Name	A.S.T.M. Partial Immersion[a]		A.S.T.M. Acid Heat[b]	
A.S.T.M. Designation	E 1 (3C – 39)	E 1 (3F – 39)	E 1 (4C – 39)	E 1 (4F – 39)
Liquid	mercury	mercury	mercury	mercury
Filling above Liquid	nitrogen gas	nitrogen gas	nitrogen gas	nitrogen gas
Temperature Range	−5 to +400 C.	+20 to 760 F.	0 to 105 C.	30 to 220 F.
Subdivisions	1 C.	2 F.	0.5 C.	1 F.
Total Length	404 to 408 mm. (15.91 to 16.06 in.)		378 to 384 mm. (14.88 to 15.12 in.)	
Stem Diameter	6.0 to 7.0 mm. (0.236 to 0.276 in.)		6.0 to 7.0 mm. (0.236 to 0.276 in.)	
Bulb Diameter	5.0 to 6.0 mm. (0.197 to 0.236 in.)		5.0 to 6.0 mm. (0.197 to 0.236 in.)	
Bulb Length	10 to 15 mm. (0.39 to 0.59 in.)		10 to 15 mm. (0.39 to 0.59 in.)	
Bottom of Bulb to Graduation Line at Distance	0 C. 100 to 110 mm. (3.94 to 4.33 in.)	32 F.	0 C. 200 to 215 mm. (7.87 to 8.46 in.)	32 F.
Top of Thermometer to Graduation Line at Distance	400 C. 25 to 50 mm. (0.98 to 1.97 in.)	752 F.	105 C. 25 to 40 mm. (0.98 to 1.57 in.)	220 F.
Top Finish	glass ring	glass ring	glass ring	glass ring
Longer Graduation Lines at Each	5 C.	10 F.	1 C.	5 F.
Graduations Numbered at Each Multiple of	10 C.	20 F.	5 C.	10 F.
Immersion	76 mm.[c]	3 in.[c]	152 mm.[e]	6 in.[e]
Special Marking on Thermometer (See Section 7)	ASTM 76-mm. Imm.	ASTM 3-in. Imm.	ASTM Acid Heat 152-mm. Imm.	ASTM Acid Heat 6-in. Imm.
Scale Error at any point when standardized shall not exceed	{1 C. up to 301 C. {1.5 C. over 301 C.	{2 F. up to 574 F. {3 F. over 574 F.	0.5 C.	1 F.
Standardization	*d*	*d*	*f*	*f*
Test for Permanency of Range (See Section 10)	subject to 10 C. below max. scale temp. for 24 hr	subject to 20 F. below max. scale temp. for 24 hr.	....	....
Marking on Case	A.S.T.M. −5 to +400 C., 76-mm. immersion	A.S.T.M. 20 to 760 F., 3-in. immersion	A.S.T.M. Acid Heat, 0 to 105 C.	A.S.T.M. Acid Heat, 30 to 220 F.

[a] These requirements were previously specified in A.S.T.M. Standard D 184.
[b] These requirements were previously specified in A.S.T.M. Method D 481.
[c] A line around the stem 76 mm. or 3 in. above the bottom of the bulb shall be etched on the thermometer.
[d] The thermometer shall be standardized for 76-mm. or 3-in. immersion and for the following temperatures of the emergent mercury column. These stem temperatures have been chosen as corresponding, on the average, to those likely to occur in the use of the thermometer.

Thermometer Reading	Average Temperature of Emergent Mercury Column	Thermometer Reading	Average Temperature of Emergent Mercury Column
100 C	50 C.	212 F	122 F.
200 C	75 C.	392 F	167 F.
300 C	89 C.	572 F	192 F.
400 C	94 C.	752 F	201 F.

[e] A line around the stem 152 mm. or 6 in. above the bottom of the bulb shall be etched on the thermometer.
[f] The thermometer shall be standardized at the ice point and at intervals of approximately 30 C. or 50 F. for 152-mm or 6-in. immersion and for the following temperatures of the emergent mercury column:

Thermometer Reading	Average Temperature of Emergent Mercury Column	Thermometer Reading	Average Temperature of Emergent Mercury Column
20 C	20 C.	70 F	70 F.
40 C	31 C.	100 F	86 F.
70 C	40 C.	150 F	104 F.
100 C	48 C.	212 F	118 F.

TABLE I.—REQUIREMENTS FOR A.S.T.M. THERMOMETERS (*Continued*).

Name	A.S.T.M. Cloud and Pour[a]		A.S.T.M. Low Cloud and Pour[a]	
A.S.T.M. Designation	E 1 (5C – 39)	E 1 (5F – 39)	E1 (6C – 39)	E 1 (6F – 39)
Liquid	mercury	mercury	toluene[b]	toluene[b]
Filling above Liquid	nitrogen gas	nitrogen gas	gas under pressure	gas under pressure
Temperature Range	−38 to +50 C.	−36 to +120 F.	−60 to +20 C.	−70 to +70 F.
Subdivisions	1 C.	2 F.	1 C.	2 F.
Total Length	220 to 224 mm. (8.66 to 8.82 in.)		227 to 231 mm. (8.94 to 9.09 in.)	
Stem Diameter	7.0 to 8.0 mm. (0.276 to 0.315 in.)		7.0 to 8.0 mm. (0.276 to 0.315 in.)	
Bulb Diameter	not greater than stem		5.0 to 6.5 mm. (0.197 to 0.256 in.)	
Bulb Length	not over 9.5 mm. (0.374 in.)		8.0 to 9.5 mm. (0.315 to 0.374 in.)	
Bottom of Bulb to Graduation Line at Distance	−38 C. 120 to 130 mm.	−36 F. (4.72 to 5.12 in.)	−57 C. 120 to 130 mm.	−70 F. (4.72 to 5.12 in.)
Top of Thermometer to Graduation Line at Distance	+49 C. 19 to 25 mm.	+120 F. (0.75 to 0.98 in.)	+20 C. 35 to 45 mm.	+68 F. (1.38 to 1.77 in.)
Expansion Chamber shall permit heating to	+100 C.	+212 F.	+60 C.	+140 F.
Top Finish	plain	plain	plain	plain
Longer Graduation Lines at Each	5 C.	10 F.	5 C.	10 F.
Graduations Numbered at Each Multiple of	10 C.	20 F.	10 C.	20 F.
Immersion	108 mm.[c]	4¼ in.[c]	76 mm.[d]	3 in.[d]
Special Marking on Thermometer (See Section 7)	108-mm. Imm. ASTM Cloud and Pour	4¼-in. Imm. ASTM Cloud and Pour	76-mm. Imm. ASTM Low Cloud and Pour	3-in. Imm. ASTM Low Cloud and Pour
Scale Error at any point when standardized shall not exceed	0.5 C.	1 F.	1 C.	2 F.
Standardization	ice point, every 20 C.; and avg. temp. 21 C. of emergent stem	ice point, every 40 F.; and avg. temp. 70 F. of emergent stem	ice point, every 20 C.; and avg. temp. 21 C. of emergent stem	ice point, every 35 F.; and avg. temp. 70 F. of emergent stem
Marking on Case	A.S.T.M. Cloud and Pour, −38 to +50 C.	A.S.T.M. Cloud and Pour, −36 to +120 F.	A.S.T.M. Low Cloud and Pour, −60 to +20 C.	A.S.T.M. Low Cloud and Pour, −70 to +70 F.

[a] These requirements were previously specified in A.S.T.M. Method D 97.
[b] Toluene or other suitable liquid colored red with a permanent dye shall be used.
[c] A line around the stem 108 mm. or 4¼ in. above the bottom of the bulb shall be etched on the thermometer.
[d] A line around the stem 76 mm. or 3 in. above the bottom of the bulb shall be etched on the thermometer.

TABLE I.—REQUIREMENTS FOR A.S.T.M. THERMOMETERS (*Continued*).

Name	A.S.T.M. Low Distillation[a]		A.S.T.M. High Distillation[b]	
A.S.T.M. Designation	E 1 (7C – 39)	E 1 (7F – 39)	E 1 (8C – 42)	E 1 (8F – 42)
Liquid	mercury	mercury	mercury	mercury
Filling above Liquid	nitrogen gas	nitrogen gas	nitrogen gas	nitrogen gas
Temperature Range	0 to 300 C.	30 to 580 F.	0 to 400 C.[c]	30 to 760 F.[c]
Subdivisions	1 C.[d]	2 F.	1 C.[d]	2 F.
Total Length	378 to 384 mm. (14.88 to 15.12 in.)		378 to 384 mm. (14.88 to 15.12 in.)	
Stem Diameter	6.0 to 7.0 mm. (0.236 to 0.276 in.)		6.0 to 7.0 mm. (0.236 to 0.276 in.)	
Bulb Diameter	5.0 to 6.0 mm. (0.197 to 0.236 in.)		5.0 to 6.0 mm. (0.197 to 0.236 in.)	
Bulb Length	10 to 15 mm. (0.39 to 0.59 in.)		10 to 15 mm. (0.39 to 0.59 in.)	
Bottom of Bulb to Graduation Line at..... Distance.....	0 C. 100 to 110 mm. (3.94 to 4.33 in.)	32 F.	0 C. 25 to 45 mm. (0.98 to 1.77 in.)	32 F.
Top of Thermometer to Graduation Line at..... Distance.....	300 C. 30 to 45 mm. (1.18 to 1.77 in.)	572 F.	400 C. 30 to 45 mm. (1.18 to 1.77 in.)	752 F.
Top Finish	glass ring	glass ring	glass ring	glass ring
Longer Graduation Lines at Each	5 C.	10 F.	5 C.	10 F.
Graduations Numbered at Each Multiple of	10 C.	20 F.	10 C.	20 F.
Immersion	total	total	total	total
Special Marking on Thermometer (See Section 7)	ASTM Low Distillation	ASTM Low Distillation	ASTM High Distillation	ASTM High Distillation
Scale Error at any point up to..... when standardized, shall not exceed	300 C. 0.5 C.	572 F. 1 F.	370 C. 1 C.	700 F. 2 F.
Standardization	ice point, every 50 C., and at 300 C.	ice point, every 100 F., and at 572 F.	ice point, every 50 C., and at 370 C.	ice point, every 100 F., and at 700 F.
Test for Permanency of Range (See Section 10).	subject to 280 to 290 C. for 24 hr.	subject to 540 to 560 F. for 24 hr.	subject to 360 to 370 C. for 24 hr.	subject to 680 to 700 F. for 24 hr.
Marking on Case	A.S.T.M. Low Distillation, 0 to 300 C.	A.S.T.M. Low Distillation, 30 to 580 F.	A.S.T.M. High Distillation, 0 to 400 C.	A.S.T.M. High Distillation, 30 to 760 F.

[a] These requirements were previously specified in A.S.T.M. Methods D 86, D 216, and D 285.

[b] These requirements were previously specified in A.S.T.M. Methods D 20, D 86, D 246, D 370, and D 402, except for the revision adopted in 1942.

[c] Under certain test conditions, the bulb of the thermometer may be 28 C. (50 F.) above the temperature indicated by the thermometer, and at an indicated temperature of 371 C. (700 F.) the temperature of the bulb is approaching a critical range in the glass. It is therefore not desirable to use this thermometer under such conditions at indicated temperatures above 371 C. (700 F.) without checking the ice point.

[d] For purposes of calibration and certification two or three division markings should be placed below the zero on the Centigrade scale thermometer.

TABLE I.—REQUIREMENTS FOR A.S.T.M. THERMOMETERS (*Continued*).

Name	A.S.T.M. Pensky-Martens, Low Range[a] A.S.T.M. Tag Closed Tester[a]		A.S.T.M. Pensky-Martens, High Range[b]	
A.S.T.M. Designation	E 1 (9C - 39)	E 1 (9F - 39)	E 1 (10C - 39)	E 1 (10F - 39)
Liquid	mercury	mercury	mercury	mercury
Filling above Liquid	nitrogen gas	nitrogen gas	nitrogen gas	nitrogen gas
Temperature Range	−7 to +110 C.	+20 to +230 F.	90 to 370 C.	200 to 700 F.
Subdivisions	0.5 C.	1 F.	2 C.	5 F.
Total Length	273 to 277 mm. (10.75 to 10.91 in.)		273 to 277 mm. (10.75 to 10.91 in.)	
Stem Diameter	6.0 to 7.0 mm. (0.236 to 0.276 in.)		6.0 to 7.0 mm. (0.236 to 0.276 in.)	
Bulb Diameter	not greater than stem		not greater than stem	
Bulb Length	9.0 to 13 mm. (0.354 to 0.51 in.)		not over 10 mm. (0.39 in.)	
Bottom of Bulb to Graduation Line at Distance	−7 C. 75 to 90 mm. (2.95 to 3.54 in.)	+20 F. 2.95 to 3.54 in.)	93 C. 75 to 90 mm. (2.95 to 3.54 in.)	200 F. 2.95 to 3.54 in.)
Top of Thermometer to Graduation Line at Distance	+110 C. 25 to 40 mm. (0.98 to 1.57 in.)	+230 F. 0.98 to 1.57 in.)	371 C. 25 to 40 mm. (0.98 to 1.57 in.)	700 F. 0.98 to 1.57 in.)
Expansion Chamber	[c]		....	....
Top Finish	glass ring	glass ring	glass ring	glass ring
Longer Graduation Lines at Each	1 C.	5 F.	10 C.	25 F.
Graduations Numbered at Each Multiple of	5 C.	10 F.	20 C.	50 F.
Immersion	57 mm.[d]	2¼ in.[d]	57 mm.[d]	2¼ in.[d]
Special Marking on Thermometer (See Section 7)	57-mm. Imm. ASTM P.M. and Tag	2¼-in. Imm. ASTM P.M. and Tag	57-mm. Imm. ASTM P.M. High	2¼-in. Imm. ASTM P.M. High
Scale Error at any point when standardized shall not exceed	0.5 C.	1 F.	1 C.	2.5 F.
Standardization	[e]	[e]	[f]	[f]
Test for Permanency of Range (See Section 10)	....	....	subject to 360 to 370 C., for 24 hr.	subject to 680 to 700 F., for 24 hr.
Marking on Case	A.S.T.M. P.M. and Tag, −7 to +110 C.	A.S.T.M. P.M. and Tag, +20 to +230 F.	A.S.T.M. P.M. High, 90 to 370 C.	A.S.T.M. P.M. High, 200 to 700 F.

[a] These requirements were previously specified in A.S.T.M. Methods D 56 and D 93.
[b] These requirements were previously specified in A.S.T.M. Method D 93.
[c] The expansion chamber shall permit heating the thermometer at least 50 C. or 90 F. above the highest temperature on the scale.
[d] A line around the stem 57 mm. or 2¼ in. above the bottom of the bulb shall be etched on the thermometer.
[e] The thermometer shall be standardized at the ice point and at intervals of approximately 30 C. or 50 F. for 57-mm. or 2¼-in. immersion and for the following temperatures of the emergent mercury column:

Thermometer Reading	Average Temperature of Emergent Mercury Column	Thermometer Reading	Average Temperature of Emergent Mercury Column
20 C	20 C.	70 F	70 F.
40 C	31 C.	100 F	86 F.
70 C	40 C.	150 F	104 F.
100 C	48 C.	212 F	118 F.

The thermometer shall be standardized at intervals of approximately 50 C. or 100 F. for 57-mm. or 2¼-in. immersion and for the following temperatures of the emergent mercury column:

Thermometer Reading	Average Temperature of Emergent Mercury Column	Thermometer Reading	Average Temperature of Emergent Mercury Column
100 C	61 C.	200 F	140 F.
150 C	65 C.	300 F	149 F.
200 C	71 C.	400 F	160 F.
250 C	78 C.	500 F	175 F.
300 C	87 C.	600 F	195 F.
350 C	99 C.	700 F	220 F.

[f] These requirements were previously specified in A.S.T.M. Methods D 56 and D 93.

TABLE I.—REQUIREMENTS FOR A.S.T.M. THERMOMETERS (*Continued*).

Name	A.S.T.M. Open Flash[a]		A.S.T.M. Gravity[b]	
A.S.T.M. Designation	E 1 (11C – 42)	E 1 (11F – 42)	E 1 (12C – 39)	E 1 (12F – 39)
Liquid	mercury	mercury	mercury	mercury
Filling above Liquid	nitrogen gas	nitrogen gas	nitrogen gas	nitrogen gas
Temperature Range	−6 to +400 C.	+20 to +760 F.	−20 to +102 C.	−5 to +215 F.
Subdivisions	2 C.	5 F.	0.2 C.	0.5 F.
Total Length	303 to 307 mm. (11.93 to 12.09 in.)		403 to 409 mm. (15.87 to 16.10 in.)	
Stem Diameter	6.0 to 7.0 mm. (0.236 to 0.276 in.)		7.0 to 8.0 mm. (0.276 to 0.315 in.)	
Bulb Diameter	not greater than stem		6.0 to 7.0 mm. (0.236 to 0.276 in.)	
Bulb Length	not over 10 mm. (0.39 in.)		14 to 19 mm. (0.55 to 0.75 in.)	
Bottom of Bulb to Graduation Line at Distance	−6 C. 40 to 50 mm. (1.57 to 1.97 in.)	+20 F.	−20 C. 32 to 51 mm. (1.26 to 2.01 in.)	−5 F.
Top of Thermometer to Graduation Line at Distance	+400 C. 30 to 45 mm. (1.18 to 1.77 in.)	+760 F.	+102 C. 25 to 45 mm. (0.98 to 1.77 in.)	+215 F.
Top Finish	red glass ring	red glass ring	glass ring	glass ring
Longer Graduation Lines at Each	10 C.	10 F.	1 C.	1 F.
Graduations Numbered at Each Multiple of	10 C.	20 F.	2 C.	5 F.
Immersion	25 mm.[c]	1 in.[c]	total	total
Special Marking on Thermometer (See Section 7).	25-mm. Imm. ASTM Open Flash	1-in. Imm. ASTM Open Flash	ASTM Gr.	ASTM Gr.
Scale Error at any point up to when standardized, shall not exceed	372 C. 1 C.	700 F. 2.5 F.	0.1 C.	0.25 F.
Standardization	[d]	[d]	ice point and every 15 or 20 C.	ice point and every 30 or 40 F.
Test for Permanency of Range (See Section 10)	subject to 360 to 370 C. for 24 hr.	subject to 680 to 700 F. for 24 hr.	....	....
Marking on Case	A.S.T.M. Open Flash, −6 to +400 C.	A.S.T.M. Open Flash, +20 to +760 F.	A.S.T.M. Gr., −20 to +102 C.	A.S.T.M. Gr., −5 to +215 F.

[a] These requirements were previously specified in A.S.T.M. Methods D 92 and D 243, except for the revision adopted in 1942.

[b] These requirements were previously specified in A.S.T.M. Method D 287.

[c] A line around the stem 25 mm. or 1 in. above the bottom of the bulb shall be etched on the thermometer.

[d] The thermometer shall be standardized at the ice point and at intervals of approximately 50 C. or 100 F. for 25-mm. or 1-in. immersion and for the following temperatures of the emergent mercury column:

Thermometer Reading	Average Temperature of Emergent Mercury Column	Thermometer Reading	Average Temperature of Emergent Mercury Column
100 C	44 C.	200 F	110 F.
150 C	54 C.	300 F	129 F.
200 C	64 C.	400 F	150 F.
250 C	77 C.	500 F	175 F.
300 C	91 C.	600 F	205 F.
350 C	108 C.	700 F	240 F.

TABLE I.—REQUIREMENTS FOR A.S.T.M. THERMOMETERS (*Continued*).

Name	A.S.T.M. Loss on Heat[a]	A.S.T.M. Paraffin Wax Melting Point[b]	
A.S.T.M. Designation	E 1 (13C – 39)	E 1 (14C – 39)	E 1 (14F – 39)
Liquid	mercury	mercury	mercury
Filling above Liquid	nitrogen gas	nitrogen gas	nitrogen gas
Temperature Range	155 to 170 C.	38 to 82 C.	100 to 180 F.
Subdivisions	0.5 C.	0.1 C.	0.2 F.
Total Length	150 to 154 mm. (5.91 to 6.06 in.)	365 to 371 mm. (14.37 to 14.61 in.)	
Stem Diameter	6.0 to 7.0 mm. (0.236 to 0.276 in.)	6.0 to 7.0 mm. (0.236 to 0.276 in.)	
Bulb Diameter	5.0 to 6.0 mm. (0.197 to 0.236 in.)	not greater than stem	
Bulb Length	10 to 15 mm. (0.39 to 0.59 in.)	not over 28 mm. (1.10 in.)	not over 28 mm. (1.10 in.)
Bottom of Bulb to Graduation Line at Distance	0 C.[g] 33 to 38 mm. (1.30 to 1.50 in.) / 155 C. 70 to 80 mm. (2.76 to 3.15 in.)	38 C. 105 to 115 mm. (4.13 to 4.53 in.)	100 F.
Top of Thermometer to Graduation Line at Distance	170 C. 20 to 30 mm. (0.79 to 1.18 in.)	82 C. 25 to 40 mm. (0.98 to 1.57 in.)	180 F.
Contraction Chamber	[c]	[d]	[d]
Expansion Chamber shall permit heating to	[e]	100 C.	212 F.
Top Finish	glass ring	plain	plain
Longer Graduation Lines at Each	1 C.	0.5 C.	1.0 F.
Graduations Numbered at Each Multiple of	5 C.; and at 0 C. and 163 C.	1 C.	2 F.
Immersion	total	79 mm.[f]	3 1/8 in.[f]
Special Marking on Thermometer (See Section 7)	ASTM Loss on Heat	ASTM Pfe. M.P. 79-mm. Imm.	ASTM Pfe. M.P. 3 1/8-in. Imm.
Scale Error at any point when standardized shall not exceed	0.5 C.	0.1 C.	0.2 F.
Standardization	at three points including 163 C. for total immersion	every 10 C. for 79-mm. immersion, and for avg. temp. 25 C. of emergent stem	every 20 F. for 3 1/8-in. immersion, and for avg. temp. 77 F. of emergent stem
Marking on Case	A.S.T.M. Loss on Heat, 155 to 170 C.	A.S.T.M. Pfe. M.P., 38 to 82 C.	A.S.T.M. Pfe. M.P., 100 to 180 F.

[a] These requirements were previously specified in A.S.T.M. Method D 6, except for the editorial changes made in 1942.
[b] These requirements were previously specified in A.S.T.M. Methods D 87 and D 127.
[c] The contraction chamber shall be of the long, narrow type, the top shall be not more than 65 mm. (2.56 in.) above the bottom of the bulb.
[d] The top of the contraction chamber shall be not more than 41 mm. (1.61 in.) above the bottom of the bulb.
[e] The expansion chamber shall permit heating the thermometer at least 25 C. above the highest temperature on the scale.
[f] A line around the stem 79 mm. or 3 1/8 in. above the bottom of the bulb shall be etched on the thermometer.
[g] At the ice point the thermometer should be graduated from − 1 C. to + 1 C. in 0.5 C. divisions.

TABLE I.—REQUIREMENTS FOR A.S.T.M. THERMOMETERS (*Continued*).

Name	A.S.T.M. Low Softening Point[a]		A.S.T.M. High Softening Point[b]	
A.S.T.M. Designation	E 1 (15C – 39)	E 1 (15F – 39)	E 1 (16C – 39)	E 1 (16F – 39)
Liquid	mercury	mercury	mercury	mercury
Filling above Liquid	nitrogen gas	nitrogen gas	nitrogen gas	nitrogen gas
Temperature Range	−2 to +80 C.	+30 to +180 F.	30 to 200 C.	85 to 392 F.
Subdivisions	0.2 C.	0.5 F.	0.5 C.	1 F.
Total Length	378 to 384 mm. (14.88 to 15.12 in.)		378 to 384 mm. (14.88 to 15.12 in.)	
Stem Diameter	6.0 to 7.0 mm. (0.236 to 0.276 in.)		6.0 to 7.0 mm. (0.236 to 0.276 in.)	
Bulb Diameter	4.5 to 5.5 mm. (0.177 to 0.217 in.)		4.5 to 5.5 mm. (0.177 to 0.217 in.)	
Bulb Length	9.0 to 14 mm. (0.354 to 0.55 in.)		9.0 to 14 mm. (0.354 to 0.55 in.)	
Bottom of Bulb to Graduation Line at Distance	0 C. 75 to 90 mm. (2.95 to 3.54 in.)	32 F.	30 C. 75 to 90 mm. (2.95 to 3.54 in.)	85 F.
Top of Thermometer to Graduation Line at Distance	80 C. 30 to 45 mm. (1.18 to 1.77 in.)	176 F.	200 C. 30 to 45 mm. (1.18 to 1.77 in.)	392 F.
Expansion Chamber shall permit heating to at least	130 C.	270 F.	250 C.	482 F.
Top Finish	glass ring	glass ring	glass ring	glass ring
Longer Graduation Lines at Each	1 C.	1 F.	1 C.	5 F.
Graduations Numbered at Each Multiple of	2 C.	5 F.	5 C.	10 F.
Immersion	total	total	total	total
Special Marking on Thermometer (See Section 7)	ASTM Low S.P.	ASTM Low S.P.	ASTM High S.P.	ASTM High S.P.
Scale Error at any point when standardized shall not exceed	0.2 C.	0.4 F.	0.3 C.	0.5 F.
Standardization	ice point, and every 20 C.	ice point, and every 40 F.	every 40 C.	every 70 F.
Marking on Case	A.S.T.M. Low S.P., −2 to +80 C.	A.S.T.M. Low S.P., +30 to +180 F.	A.S.T.M. High S.P., 30 to 200 C.	A.S.T.M. High S.P., 85 to 392 F.

[a] These requirements were previously specified in A S.T.M. Methods D 36, D 61, D 139, and E 28.
[b] These requirements were previously specified in A.S.T M. Method D 36, except for the temperature range, and in Method E 28.

TABLE I.—REQUIREMENTS FOR A.S.T.M. THERMOMETERS (*Continued*).

Name	A.S.T.M. Saybolt Viscosity[a]		A.S.T.M. Saybolt Viscosity	A.S.T.M. Saybolt Viscosity[a] A.S.T.M. Reid Vapor Pressure[g]
A.S.T.M. Designation	E 1 (17C – 39)	E 1 (17F – 39)	E 1 (18C – 39)	E 1 (18F – 39)
Liquid	mercury	mercury	mercury	mercury
Filling above Liquid	nitrogen gas	nitrogen gas	nitrogen gas	nitrogen gas
Temperature Range, For Tests at	19 to 27 C. 21.1 and 25 C.	66 to 80 F. 70 and 77 F.	34 to 42 C. 37.8 C.	94 to 108 F. 100 F.
Subdivisions	0.1 C.	0.2 F.	0.1 C.	0.2 F.
Total Length	252 to 256 mm. (9.92 to 10.08 in.)		252 to 256 mm. (9.92 to 10.08 in.)	
Stem Diameter[f]	6.0 to 7.0 mm. (0.236 to 0.276 in.)		6.0 to 7.0 mm. (0.236 to 0.276 in.)	
Bulb Diameter	not less than 5.0 mm. (0.197 in.) not greater than that of stem		not less than 5.0 mm. (0.197 in.) not greater than that of stem	
Bulb Length	25 to 35 mm. (0.98 to 1.38 in.)		25 to 35 mm. (0.98 to 1.38 in.)	
Bottom of Bulb to Graduation Line at, Distance	19 C. 135 to 150 mm.	66 F. (5.32 to 5.91 in.)	34 C. 135 to 150 mm.	94 F. (5.32 to 5.91 in.)
Top of Thermometer to Graduation Line at, Distance	27 C. 20 to 35 mm.	80 F. (0.79 to 1.38 in.)	42 C. 20 to 35 mm.	108 F. (0.79 to 1.38 in.)
Contraction Chamber	[b]	[b]	[b]	[b]
Expansion Chamber	[c]	[c]	[c]	[c]
Top Finish	glass ring	glass ring	glass ring	glass ring
Longer Graduation Lines at Each	0.5 C.	1 F.	0.5 C.	1 F.
Graduations Numbered at Each Multiple of	1 C.	2 F.	1 C.	2 F.
Immersion	total	total	total	total
Special Marking on Thermometer (See Section 7)	ASTM 21.1 and 25 C.[d]	ASTM 70 and 77 F.[d]	ASTM 37.8 C.[d]	ASTM 100 F.[d]
Scale Error at any point shall not exceed	0.1 C.	0.2 F.	0.1 C.	0.2 F.
Standardization	total immersion[e]	total immersion[e]	total immersion[e]	total immersion[e]
Marking on Case	A.S.T.M. Saybolt Viscosimeter Thermometer, 19 to 27 C.	A.S.T.M. Saybolt Viscosimeter Thermometer, 66 to 80 F.	A.S.T.M. Saybolt Viscosimeter Thermometer, 34 to 42 C.	A.S.T.M. Saybolt Viscosimeter Thermometer, 94 to 108 F. or A.S.T.M. Reid Vapor Pressure Thermometer

[a] These requirements were previously specified in A.S.T.M.Method D 88.

[b] The contraction chamber shall be of the long, narrow type; the top shall be not more than 60 mm. (2.36 in.) above the bottom of the bulb. The mercury shall stand in the contraction chamber at 0 C. (32 F.).

[c] The expansion chamber shall permit heating the thermometer 50 C. (90 F.) above the highest temperature on the scale, and in all cases shall permit heating to 100 C. (212 F.).

[d] The test temperatures shall be shown in full at the appropriate points on the scale and the graduations corresponding to these points and the numbers shall be in red.

[e] Correction for emergent stem shall not be applied.

[f] The stem shall be made with an enlargement not less than 4.0 nor more than 7.0 mm. (0.157 to 0.276 in.) in length, having a diameter 2.0 to 3.0 mm. (0.079 to 0.118 in.) greater than that of the stem, the bottom of the enlargement being 114 mm. (4.49 in.) above the bottom of the bulb.

[g] These requirements were previously specified in A.S.T.M. Method D 323.

TABLE I.—REQUIREMENTS FOR A.S.T.M. THERMOMETERS (*Continued*).

Name	A.S.T.M. Saybolt Viscosity[a]		A.S.T.M. Saybolt Viscosity[a]	
A.S.T.M. Designation	F 1 (19C – 39)	E 1 (19F – 39)	E 1 (20C – 39)	E 1 (20F – 39)
Liquid	mercury	mercury	mercury	mercury
Filling above Liquid	nitrogen gas	nitrogen gas	nitrogen gas	nitrogen gas
Temperature Range For Tests at	49 to 57 C. 50 and 54.4 C.	120 to 134 F. 122 and 130 F.	57 to 65 C. 60 C.	134 to 148 F. 140 F.
Subdivisions	0.1 C.	0.2 F.	0.1 C.	0.2 F.
Total Length	252 to 256 mm. (9.92 to 10.08 in.)		252 to 256 mm. (9.92 to 10.08 in.)	
Stem Diameter[f]	6.0 to 7.0 mm. (0.236 to 0.276 in.)		6.0 to 7.0 mm. (0.236 to 0.276 in.)	
Bulb Diameter	not less than 5.0 mm. (0.197 in.) not greater than that of stem		not less than 5.0 mm. (0.197 in.) not greater than that of stem	
Bulb Length	25 to 35 mm. (0.98 to 1.38 in.)		25 to 35 mm. (0.98 to 1.38 in.)	
Bottom of Bulb to Graduation Line at Distance	49 C. 135 to 150 mm.	120 F. (5.32 to 5.91 in.)	57 C. 135 to 150 mm.	134 F. (5.32 to 5.91 in.)
Top of Thermometer to Graduation Line at Distance	57 C. 20 to 35 mm.	134 F. (0.79 to 1.38 in.)	65 C. 20 to 35 mm.	148 F. (0.79 to 1.38 in.)
Contraction Chamber	[b]	[b]	[b]	[b]
Expansion Chamber	[c]	[c]	[c]	[c]
Top Finish	glass ring	glass ring	glass ring	glass ring
Longer Graduation Lines at Each	0.5 C.	1 F.	0.5 C.	1 F.
Graduations Numbered at Each Multiple of	1 C.	2 F.	1 C.	2 F.
Immersion	total	total	total	total
Special Marking on Thermometer (See Section 7)	ASTM 50 and 54.4 C.[d]	ASTM 122 and 130 F.[d]	ASTM 60 C.[d]	ASTM 140 F.[d]
Scale Error at any point shall not exceed	0.1 C.	0.2 F.	0.1 C.	0.2 F.
Standardization	total immersion[e]	total immersion[e]	total immersion[e]	total immersion[e]
Marking on Case	A.S.T.M. Saybolt Viscosimeter Thermometer, 49 to 57 C.	A.S.T.M. Saybolt Viscosimeter Thermometer, 120 to 134 F.	A.S.T.M. Saybolt Viscosimeter Thermometer, 57 to 65 C.	A.S.T.M. Saybolt Viscosimeter Thermometer, 134 to 148 F.

[a] These requirements were previously specified in A.S.T.M. Standard Method D 88.

[b] The contraction chamber shall be of the long, narrow type; the top shall be not more than 60 mm. (2.36 in.) above the bottom of the bulb. The mercury shall stand in the contraction chamber at 0 C. (32 F.).

[c] The expansion chamber shall permit heating the thermometer 50 C. (90 F.) above the highest temperature on the scale, and in all cases shall permit heating to 100 C. (212 F.).

[d] The test temperatures shall be shown in full at the appropriate points on the scale and the graduations corresponding to these points and the numbers shall be in red.

[e] Correction for emergent stem shall not be applied.

[f] The stem shall be made with an enlargement not less than 4.0 nor more than 7.0 mm. (0.157 to 0.276 in.) in length, having a diameter 2.0 to 3.0 mm. (0.079 to 0.118 in.) greater than that of the stem, the bottom of the enlargement being 114 mm. (4.49 in.) above the bottom of the bulb.

TABLE I.—REQUIREMENTS FOR A.S.T.M. THERMOMETERS (*Continued*).

Name	A.S.T.M. Saybolt Viscosity[a]		A.S.T.M. Saybolt Viscosity[a]	
A.S.T.M. Designation	E 1 (21C – 39)	E 1 (21F – 39)	E 1 (22C – 39)	E 1 (22F – 39)
Liquid	mercury	mercury	mercury	mercury
Filling above Liquid	nitrogen gas	nitrogen gas	nitrogen gas	nitrogen gas
Temperature Range For Tests at	79 to 87 C. 82.2 C.	174 to 188 F. 180 F.	95 to 103 C. 98.9 C.	204 to 218 F. 210 F.
Subdivisions	0.1 C.	0.2 F.	0.1 C.	0.2 F.
Total Length	252 to 256 mm. (9.92 to 10.08 in.)		252 to 256 mm. (9.92 to 10.08 in.)	
Stem Diameter[f]	6.0 to 7.0 mm. (0.236 to 0.276 in.)		6.0 to 7.0 mm. (0.236 to 0.276 in.)	
Bulb Diameter	not less than 5.0 mm. (0.197 in.) not greater than that of stem		not less than 5.0 mm. (0.197 in.) not greater than that of stem	
Bulb Length	25 to 35 mm. (0.98 to 1.38 in.)		25 to 35 mm. (0.98 to 1.38 in.)	
Bottom of Bulb to Graduation Line at Distance	79 C. 135 to 150 mm. (5.32 to 5.91 in.)	174 F.	95 C. 135 to 150 mm. (5.32 to 5.91 in.)	204 F.
Top of Thermometer to Graduation Line at Distance	87 C. 20 to 35 mm. (0.79 to 1.38 in.)	188 F.	103 C. 20 to 35 mm. (0.79 to 1.38 in.)	218 F.
Contraction Chamber	[b]	[b]	[b]	[b]
Expansion Chamber	[c]	[c]	[c]	[c]
Top Finish	glass ring	glass ring	glass ring	glass ring
Longer Graduation Lines at Each	0.5 C.	1 F.	0.5 C.	1 F.
Graduations Numbered at Each Multiple of	1 C.	2 F.	1 C.	2 F.
Immersion	total	total	total	total
Special Marking on Thermometer (See Section 7).	ASTM 82.2 C.[d]	ASTM 180 F.[d]	ASTM 98.9 C.[d]	ASTM 210 F.[d]
Scale Error at any point shall not exceed	0.1 C.	0.2 F.	0.1 C.	0.2 F.
Standardization	total immersion[e]	total immersion[e]	total immersion[e]	total immersion[e]
Marking on Case	A.S.T.M. Saybolt Viscosimeter Thermometer, 79 to 87 C.	A.S.T.M. Saybolt Viscosimeter Thermometer, 174 to 188 F.	A.S.T.M. Saybolt Viscosimeter Thermometer, 95 to 103 C.	A.S.T.M. Saybolt Viscosimeter Thermometer, 204 to 218 F.

[a] These requirements were previously specified in A.S.T.M. Standard Method D 88.

[b] The contraction chamber shall be of the long, narrow type; the top shall be not more than 60 mm. (2.36 in.) above the bottom of the bulb. The mercury shall stand in the contraction chamber at 0 C. (32 F.).

[c] The expansion chamber shall permit heating the thermometer 50 C. (90 F.) above the highest emperature on the scale, and in all cases shall permit heating to 100 C. (212 F.).

[d] The test temperatures shall be shown in full at the appropriate points on the scale and the graduations corresponding to these points and the numbers shall be in red.

[e] Correction for emergent stem shall not be applied.

[f] The stem shall be made with an enlargement not less than 4.0 nor more than 7.0 mm. (0.157 to 0.276 in.) in length, having a diameter 2.0 to 3.0 mm. (0.079 to 0.118 in.) greater than that of the stem the bottom of the enlargement being 114 mm. (4.49 in.) above the bottom of the bulb.

TABLE I.—REQUIREMENTS FOR A.S.T.M. THERMOMETERS (*Continued*).

Name	A.S.T.M. Engler Viscosity[a,b]	A.S.T.M. Engler Viscosity[a,b]	A.S.T.M. Engler Viscosity[a,b]
A.S.T.M. Designation	E 1 (23C - 39)	E 1 (24C - 39)	E 1 (25C - 39)
Liquid	mercury	mercury	mercury
Filling above Liquid	nitrogen gas	nitrogen gas	nitrogen gas
Temperature Range	18 to 28 C.	39 to 54 C.	95 to 105 C.
Subdivisions	0.2 C.	0.2 C.	0.2 C.
Total Length	200 to 207 mm. (7.87 to 8.15 in.)	225 to 232 mm. (8.86 to 9.13 in.)	200 to 207 mm. (7.87 to 8.15 in.)
Stem Diameter	5.5 to 6.5 mm. (0.217 to 0.256 in.)	5.5 to 6.5 mm. (0.217 to 0.256 in.)	5.5 to 6.5 mm. (0.217 to 0.256 in.)
Bulb Diameter	5.5 to 6.5 mm. (0.217 to 0.256 in.)	5.5 to 6.5 mm. (0.217 to 0.256 in.)	5.5 to 6.5 mm. (0.217 to 0.256 in.)
Bulb Length	13 to 19 mm. (0.51 to 0.75 in.)	13 to 19 mm. (0.51 to 0.75 in.)	13 to 19 mm. (0.51 to 0.75 in.)
Bottom of Bulb to Graduation Line at Distance	18 C. 108 to 118 mm. (4.25 to 4.65 in.)	39 C. 108 to 118 mm. (4.25 to 4.65 in.)	95 C. 108 to 118 mm. (4.25 to 4.65 in.)
Top of Thermometer to Graduation Line at Distance	28 C. 30 to 40 mm. (1.18 to 1.57 in.)	54 C. 30 to 40 mm. (1.18 to 1.57 in.)	105 C. 30 to 40 mm. (1.18 to 1.57 in.)
Contraction Chamber	[c]	[c]	[c]
Expansion Chamber	[d]	[d]	[d]
Top Finish	glass button	glass button	glass button
Longer Graduation Lines at Each	1 C.	1 C.	1 C.
Graduations Numbered at Each Multiple of	2 C.	2 C.	2 C.
Immersion	90 mm. (3.54 in.)[f]	90 mm. (3.54 in.)[f]	90 mm. (3.54 in.)[f]
Special Marking on Thermometer (See Section 7).	Full figures at 25 C. ASTM Engler[e] 90-mm. Imm.[e]	Full figures at 40 and 50 C ASTM Engler[e] 90-mm. Imm.[e]	Full figures at 100 C ASTM Engler[e] 90-mm. Imm.[e]
Scale Error at any point shall not exceed	0.1 C.	0.1 C.	0.1 C.
Standardization	90-mm. (3.54 in.) immersion	90-mm. (3.54 in.) immersion	90-mm. (3.54 in.) immersion
Marking on Case	A.S.T.M. Engler Viscosimeter Thermometer, 18 to 28 C.	A.S.T.M. Engler Viscosimeter Thermometer, 39 to 54 C.	A.S.T.M. Engler Viscosimeter Thermometer, 95 to 105 C.

[a] These requirements were previously specified in A.S.T.M. Standard D 300.

[b] The thermometer shall be mounted in a brass ferrule consisting of a tubular bushing 8.0 mm (0.315 in.) in outside diameter with a flanged head approximately 12 mm. (0.47 in.) in diameter so that the upper extremity of the 8-mm. (0.315-in.) diameter is located 90 mm. (3.54 in.) from the bottom of the bulb.

[c] The contraction chamber shall be of the long, narrow type; the top shall be not more than 60 mm. (2.36 in.) above the bottom of the bulb. The mercury shall stand approximately in the middle of the chamber at 0 C.

[d] The expansion chamber shall permit heating the thermometer 50 C. above the highest temperature on the scale and in all cases to permit heating to 100 C.

[e] To be etched on the glass stem above the brass ferrule.

[f] A line around the stem 90 mm. or 3.54 in. above the bottom of the bulb shall be etched on the thermometer.

TABLE I.—REQUIREMENTS FOR A.S.T.M. THERMOMETERS *(Continued)*.

Name	A.S.T.M. Stability Test of Soluble Nitrocellulose[a,b]	A.S.T.M. Turpentine Distillation[b]	A.S.T.M. Titer Test
A.S.T.M. Designation	E 1 (26C - 42)	E 1 (27C - 42)	E 1 (36C - 42)
Liquid	mercury	mercury	mercury
Filling above Liquid	....	nitrogen gas	nitrogen gas
Temperature Range	130 to 140 C.	147 to 182 C.	−2 to +68 C.
Subdivisions	0.1 C.	0.5 C.	0.2 C.
Total Length	18 ± ¼ in.[c]	291 to 296 mm. (11.46 to 11.65 in.)	385 to 390 mm. (15.16 to 15.35 in.)
Stem Diameter	5/16 ± 1/16 in.	6.0 to 7.0 mm. (0.236 to 0.276 in.)	6.0 to 7.0 mm. (0.236 to 0.276 in.)[d]
Bulb Diameter	....	4.0 to 5.5 mm. (0.157 to 0.217 in.)	5.5 to 7.0 mm. (0.217 to 0.276 in.)[e]
Bulb Length	2 5/8 ± ¼ in.	10 to 15 mm. (0.39 to 0.59 in.)	15 to 25 mm. (0.59 to 0.98 in.)
Bottom of Bulb to Graduation Line at Distance	130 C. 8 5/8 ± ¼ in.	147 C. 100 to 115 mm.[f] (3.94 to 4.53 in.)	−2 C. 50 to 60 mm. (1.97 to 2.36 in.)
Top of Thermometer to Graduation Line at Distance	[g] [g]	182 C. 30 to 45 mm.[f] (1.18 to 1.77 in.)	68 C. 20 to 35 mm. (0.79 to 1.38 in.)
Contraction Chamber	yes	[h]	no
Expansion Chamber	[i]	permit heating to 230 C	[j]
Top Finish	....	glass ring	glass ring
Longer Graduation Lines at Each	0.5 C.	1 C.	1 C.
Graduations Numbered at	each 1 C. and 130, 135, and 140 C.	each 2 C. (even)	each 2 C.
Immersion	total	76 mm. (3 in.)[k]	45 mm. (1¾ in.)[l]
Special Marking on Thermometer (See Section 7)	ASTM Stab N.C.	ASTM Turp Dist 76-mm. Imm.	ASTM: FAC Titer Test 45-mm. Imm.
Scale Error at any point when standardized shall not exceed	0.2 C.	0.5 C.	0.2 C.
Standardization	....	At three points including 155 C. and 175 C., at 3-in. immersion, and avg. temp. 25 C. of emergent stem.	[m]
Marking on Case	A.S.T.M. Stability Nitrocellulose Thermometer, 130 to 140 C.	A.S.T.M. Turpentine Distillation Thermometer, 147 to 182 C.	A.S.T.M.: F.A.C. Titer Test Thermometer −2 to +68 C.

[a] These requirements were previously specified in A.S.T.M. Method D 301, except for the revisions adopted in 1942.

[b] Due to the application requirements for range and construction of this thermometer, it is not practicable to include reference points. Therefore, the National Bureau of Standards will issue a report instead of a certificate for this thermometer when submitted to the Bureau for test.

[c] The thermometer shall have a white enamel back.

[d] The stem may be either the plain front or lens front type. If the thermometer is of the lens front type, the cross-section of the stem shall be such that it will pass through an 8-mm. ring gage but will not enter a 5-mm. slot gage.

[e] In no case shall the bulb diameter be greater than the diameter of the stem.

[f] The graduated portion shall be not less than 131 mm. (5.16) in.

[g] The distance from the 130 C. to the 140 C. graduation marks, that is, the scale length, shall be 4¾ ± ¼ in.

[h] The contraction chamber shall be of the long, narrow type; the top shall be not more than 40 mm. (1.57 in.) above the bottom of the bulb. The mercury shall stand near the bottom of the chamber at 0 C.

[i] An expansion chamber shall be provided to accommodate expansion up to 175 C.

[j] The expansion chamber shall permit heating to 85 C. The length of unchanged capillary between highest graduation and expansion chamber shall be 10 mm.

[k] A line around the stem 76 mm. (3 in.) above the bottom of the bulb shall be etched on the thermometer.

[l] A line around the stem 45 mm. (1¾ in.) above the bottom of the bulb shall be etched on the thermometer.

[m] The thermometer shall be standardized at the ice point and at intervals of approximately 20 C. for 45-mm. immersion and for an average stem temperature of emergent mercury column of 25 C.

TABLE I.—REQUIREMENTS FOR A.S.T.M. THERMOMETERS (*Concluded*).

Name	Air Chamber Thermometer (A.S.T.M. Reid Vapor-Pressure Test)[a]	A.S.T.M. Tung[b, g]
A.S.T.M. Designation	E 1 (31F - 44)	E 1 (32C - 44)
Liquid	mercury	mercury
Filling above Liquid	....	nitrogen gas or hydrogen gas
Temperature Range	−40 or −30 F. to +120 or +130 F.	240 to 310 C.
Subdivisions	1 F.	2 C.
Total Length	254 to 305 mm. (10.00 to 12.00 in.)	195 to 200 mm. (7.68 to 7.87 in.)
Stem Diameter	6.0 to 7.0 mm. (0.236 to 0.276 in.)	6.0 to 7.0 mm. (0.236 to 0.276 in.)
Bulb Diameter	....	not greater than stem
Bulb Length	not over 25 mm. (0.98 in.)	not over 9 mm. (0.354 in.)
Contraction Chamber	none	[c]
Expansion Chamber	yes	[d]
Enlargement of Button	....	[e]
Bottom of Bulb to Graduation Line at Distance	....	*Distance to 282 C.*, 77 to 79 mm. *Distance to 240 C.*, 46 to 52 mm. *Distance to 310 C.*, 94 to 104 mm.
Top of Thermometer to Graduation Line at Distance	....	....
Top Finish	....	plain
Longer Graduation Lines at Each	....	10 C.
Graduations Numbered at Each Multiple of	....	20 C.
Immersion	total	total
Special Marking on Thermometer (See Section 7)	....	ASTM Tung
Scale Error at any point when standardized, shall not exceed	0.5 F.	[f]
Standardization	....	250 C. 282 C. 270 C. 300 C.
Marking on Case	....	A.S.T.M. Tung Oil Thermometer, 240 to 310 C.

[a] These requirements were previously specified in A.S.T.M. Method D 323, except for the editorial changes made in 1942.

[b] These requirements in part were previously specified in A.S.T.M. Specifications D 12.

[c] The contraction chamber shall be not less than 13 mm. below 240 C.

[d] The expansion chamber or capillary extension shall permit heating to 380 C.

[e] Enlargement of the button shall be so located on the stem that the under-side of the button shall be 150 mm. from the bottom of the bulb; diameter of button shall be approximately 3 mm. greater than diameter of the stem.

[f] The error at 282 C. shall not exceed 1 C. (one half scale division) The error at other points of the scale shall not exceed 2 C. (one scale division).

[g] Due to the application requirements for range and construction of this thermometer, it is not practicable to include reference points. Therefore, the National Bureau of Standards will issue a report instead of a certificate for this thermometer when submitted to the Bureau for test.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: Z23.1-1939
AMERICAN ASSOCIATION STATE
HIGHWAY OFFICIALS STANDARD
A.A.S.H.O. NO.: M 92-42

Standard Specifications for

SIEVES FOR TESTING PURPOSES[1]

WIRE CLOTH SIEVES, ROUND-HOLE AND SQUARE-HOLE SCREENS OR SIEVES

A.S.T.M. Designation: E 11 – 39

ADOPTED, 1939.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation E 11; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These specifications cover woven-wire cloth sieves, round-hole screens (sieves) and square-hole perforated plate screens (sieves) for precision testing in the classification of materials according to size (mechanical analysis, fineness, and particle size determinations). The sieves covered by these specifications are intended for general use (Note 1). A method of calibrating woven wire cloth sieves is included as information in the Appendix.

NOTE 1.—Some industries may possibly require more restricted specifications for sieves for special testing purposes.

WOVEN WIRE CLOTH SIEVES

Sieve Cloth

2. (*a*) Wire cloth for standard sieves shall be woven (not twilled, except the cloth of the 62-, 53-, 44-, and 37-micron sieves) from brass, bronze, or other suitable wire, and shall not be coated or plated.

(*b*) The average opening between the adjacent warp and the adjacent shoot wires, taken separately, shall conform to that given in column 2 of Table I, within the "permissible variation in average opening" given in column 4. Column 3 gives the approximate equivalents in inches of the basic values in millimeters given in column 2. The average diameter of the warp and of the shoot wires, taken separately, of the cloth of any given sieve shall be within the limits given in column 6 of Table I. Column 7 gives the approximate equivalents in inches of the basic values in millimeters given in column 6. The maximum width of opening between adjacent warp or shoot wires shall not exceed the nominal width of opening by more than the "permissible variation in maximum opening" given in column 5 of Table I. An exception may be made, in the case of 8-in. sieves, if the total length of all the

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee E-1 on Methods of Testing.

[2] Prior to their present adoption as standard, these specifications were published as tentative from 1925 to 1926. They were adopted in 1926, but withdrawn and republished as tentative from 1938 to 1939, being revised in 1939.

These specifications comprise a consolidation of the former Standard Specifications for Sieves for Testing Purposes (E 11 – 26) and for Round-Hole Screens for Testing Purposes (E 17 – 36), which specifications were accordingly discontinued in 1938.

TABLE I.—NOMINAL DIMENSIONS, PERMISSIBLE VARIATIONS, AND LIMITS FOR WOVEN WIRE CLOTH OF STANDARD SIEVES.

Size or Sieve Designation	Sieve Opening		Permissible Variations in Average Opening, per cent	Permissible Variations in Maximum Opening,[a] per cent	Wire Diameter	
	mm.	in. (approx. equivalents)			mm.	in. (approx. equivalents)
(1)	(2)	(3)	(4)	(5)	(6)	(7)
Coarse Series						
(4.24-in.)**.....	107.6	4.24	±2	+3	5.6 to 9.7	0.220 to 0.380
4-in.....	101.6	4.00	±2	+3	5.6 to 9.7	0.220 to 0.380
3½-in.....	88.9	3.50	±2	+3	5.3 to 9.3	0.210 to 0.365
3-in.....	76.2	3.00	±2	+3	4.8 to 8.1	0.190 to 0.320
2½-in.....	63.5	2.50	±2	+3	4.4 to 7.1	0.175 to 0.280
(2.12-in.)**.....	53.8	2.12	±2	+3	4.1 to 6.2	0.160 to 0.245
2-in.....	50.8	2.00	±2	+3	4.1 to 6.2	0.160 to 0.245
1¾-in.....	44.4	1.75	±2	+3	3.8 to 5.7	0.150 to 0.225
1½-in.....	38.1	1.50	±2	+3	3.7 to 5.3	0.145 to 0.210
1¼-in.....	31.7	1.25	±2	+3	3.5 to 4.8	0.140 to 0.190
(1.06-in.)**.....	26.9	1.06	±3	+5	3.43 to 4.50	0.135 to 0.177
1-in.....	25.4	1.00	±3	+5	3.43 to 4.50	0.135 to 0.177
⅞-in.....	22.2	0.875	±3	+5	3.23 to 4.22	0.127 to 0.166
¾-in.....	19.1	0.750	±3	+5	3.10 to 3.91	0.122 to 0.154
⅝-in.....	15.9	0.625	±3	+5	2.74 to 3.43	0.108 to 0.135
(0.530-in.)**.....	13.4	0.530	±3	+5	2.39 to 3.10	0.094 to 0.122
½-in.....	12.7	0.500	±3	+5	2.39 to 3.10	0.094 to 0.122
7/16-in.....	11.1	0.438	±3	+5	2.23 to 2.84	0.088 to 0.112
⅜-in.....	9.52	0.375	±3	+5	2.11 to 2.59	0.083 to 0.102
5/16-in.....	7.93	0.312	±3	+5	1.85 to 2.36	0.073 to 0.093
(0.265-in.)**.....	6.73	0.265	±3	+5	1.60 to 2.11	0.063 to 0.083
¼-in. (No. 3).....	6.35	0.250	±3	+5	1.60 to 2.11	0.063 to 0.083
Fine Series[b]						
5660 micron (No. 3½).....	5.66	0.223	±3	+10	1.28 to 1.90	0.050 to 0.075
4760 micron (No. 4).....	4.76	0.187	±3	+10	1.14 to 1.68	0.045 to 0.066
4000 micron (No. 5).....	4.00	0.157	±3	+10	1.00 to 1.47	0.039 to 0.058
3360 micron (No. 6).....	3.36	0.132	±3	+10	0.87 to 1.32	0.034 to 0.052
2830 micron (No. 7).....	2.83	0.111	±3	+10	0.80 to 1.20	0.031 to 0.047
2380 micron (No. 8).....	2.38	0.0937	±3	+10	0.74 to 1.10	0.0291 to 0.0433
2000 micron (No. 10).....	2.00	0.0787	±3	+10	0.68 to 1.00	0.0268 to 0.0394
1680 micron (No. 12).....	1.68	0.0661	±3	+10	0.62 to 0.90	0.0244 to 0.0354
1410 micron (No. 14).....	1.41	0.0555	±3	+10	0.56 to 0.80	0.0220 to 0.0315
1190 micron (No. 16).....	1.19	0.0469	±3	+10	0.50 to 0.70	0.0197 to 0.0276
1000 micron (No. 18).....	1.00	0.0394	±5	+15[a]	0.43 to 0.62	0.0169 to 0.0244
840 micron (No. 20).....	0.84	0.0331	±5	+15[a]	0.38 to 0.55	0.0150 to 0.0217
710 micron (No. 25).....	0.71	0.0280	±5	+15[a]	0.33 to 0.48	0.0130 to 0.0189
590 micron (No. 30).....	0.59	0.0232	±5	+15[a]	0.29 to 0.42	0.0114 to 0.0165
500 micron (No. 35).....	0.50	0.0197	±5	+15[a]	0.26 to 0.37	0.0102 to 0.0146
420 micron (No. 40).....	0.42	0.0165	±5	+25[a]	0.23 to 0.33	0.0091 to 0.0130
350 micron (No. 45).....	0.35	0.0138	±5	+25[a]	0.20 to 0.29	0.0079 to 0.0114
297 micron (No. 50).....	0.297	0.0117	±5	+25[a]	0.170 to 0.253	0.0067 to 0.0100
250 micron (No. 60).....	0.250	0.0098	±5	+25[a]	0.149 to 0.220	0.0059 to 0.0087
210 micron (No. 70).....	0.210	0.0083	±5	+25[a]	0.130 to 0.187	0.0051 to 0.0074
177 micron (No. 80).....	0.177	0.0070	±6	+40[a]	0.114 to 0.154	0.0045 to 0.0061
149 micron (No. 100).....	0.149	0.0059	±6	+40[a]	0.096 to 0.125	0.0038 to 0.0049
125 micron (No. 120).....	0.125	0.0049	±6	+40[a]	0.079 to 0.103	0.0031 to 0.0041
105 micron (No. 140).....	0.105	0.0041	±6	+40[a]	0.063 to 0.087	0.0025 to 0.0034
88 micron (No. 170).....	0.088	0.0035	±6	+40[a]	0.054 to 0.073	0.0021 to 0.0029
74 micron (No. 200).....	0.074	0.0029	±7	+60[a]	0.045 to 0.061	0.0018 to 0.0024
62 micron (No. 230).....	0.062	0.0024	±7	+90[a]	0.039 to 0.052	0.0015 to 0.0020
53 micron (No. 270).....	0.053	0.0021	±7	+90[a]	0.035 to 0.046	0.0014 to 0.0018
44 micron (No. 325).....	0.044	0.0017	±7	+90[a]	0.031 to 0.040	0.0012 to 0.0016
37 micron (No. 400).....	0.037	0.0015	±7	+90[a]	0.023 to 0.035	0.0009 to 0.0014

** The five sieves marked in the first column with a double asterisk (**) may be used instead of the 4-in., 2-in., 1-in., ½-in., and ¼-in. sieves when it is desired to have a series of sieves nesting with the Fine Series and continuing that series with the $\sqrt{2}$:1 ratio. All of the other sieves listed above are in a $\sqrt{2}$:1 ratio with the Fine Series within the limit of the specified permissible variations. Care should be taken in designating the five sieves marked with the double asterisk; they should not be designated as 4-in., 2-in., 1-in., ½-in., and ¼-in., but as 4.24-in., 2.12-in., 1.06-in., 0.530-in., and 0.265-in. (or by the manufacturer's nominal values, for example, for the last three 1.050-in., 0.525-in., and 0.263-in).

[a] For sieves from the 1000-micron (No. 18) to the 37-micron (No. 400) size, inclusive, not more than 5 per cent of the openings shall exceed the nominal opening by more than one-half of the permissible variation in maximum opening.

[b] See Note 2, Section 2.

portions of rows of openings exceeding this maximum width is less than 4 in. in both the warp and the shoot directions, considered separately, and provided that the sieve is not rejected under Paragraph (*d*). For sieves from the 1000-micron (No. 18) to the 37-micron (No. 400) size, inclusive, not more than 5 per cent of the openings shall exceed the nominal opening by more than one-half of the permissible variation in maximum opening.

(*c*) Both the warp and shoot wires shall be crimped in such a manner that they will be rigid when in use.

(*d*) There shall be no punctures or other obvious defects in the cloth.

NOTE 2.—The micron designation of the fine sieve series represents a strong trend among users of precision sieves toward the use of the micron terminology in reporting particle sizes. The openings of successive sieves of the fine series progress in the ratio $\sqrt[4]{2}$: 1, and in selecting sieves from this series it is customary to take each sieve in a given range, every alternate sieve, or every fourth sieve.

Standard 8-in. Sieve Frames

3. (*a*) *Sieve Frames for Coarse Series.*—Sieves of the coarse series having nominal openings of less than 1 in. may have frames of the standard 8-in. size (see Section 3 (*b*)) or may have larger frames as may be specified in individual cases. Frames for sieves of the coarse series having nominal openings of 1 in. or more may be made of either hardwood or metal and may be square, rectangular, or circular, as specified, a size larger than 8-in. being recommended. Frames, covers, and pans of the standard 8-in. size shall be made of brass, unless otherwise specified.

NOTE 3: *Special Sieves.*—These specifications do not preclude the use of special sieves for special purposes, as for example sieves having a diameter other than 8 in. or the nesting sieves for field use. When such sieves are used, in place of the standard 8-in. sieve, the cloth of the sieves should be required to conform to these specifications. The use of other than standard 8-in. sieves where these standard sieves could be used should be discouraged, as the results are not necessarily comparable.

(*b*) *Sieve Frames for Fine Series.*—Frames for all sieves of the fine series shall be the standard 8-in. size, except that frames 3 in. in diameter may be used in the case of sieves No. 100 and finer, used primarily in the testing of paint pigments. The standard frames shall be circular, 8 in. (20.32 cm.) in diameter. The height of the sieve from the top of the frame to the cloth shall be either about 2 in. (5 cm.), or 1 in. (2.5 cm.). Sieves having a height of 2 in. (5 cm.) shall be designated as full-height sieves; those having a height of 1 in. (2.5 cm.) as half-height sieves. The permissible variation on the mean inside diameter $\frac{3}{16}$ in. below the top of the sieve shall be plus $\frac{3}{32}$ in. The bottom of the sieve or "sieve skirt" shall be so constructed as to have an easy sliding fit in any sieve conforming to the above permissible variations and in no case shall this outside diameter be less than 7.970 in. nor more than 8.000 in. Pans and covers shall be so made as to be interchangeable with standard sieves.

(*c*) *Mounting of Cloth in Frame.*—The cloth shall be mounted on the frame without distortion, looseness, or waviness. To prevent the material being sieved from catching in the joint between the cloth and the frame, the joint shall be smoothly filled with solder or so made that the material will not catch.

Three-Inch Sieves

4. (*a*) Sieves 3 in. in diameter, used for testing paint pigments, shall be made from standard wire cloth No. 100 or finer. The sieve frames shall be circular, about 3 in. (7.6 cm.) in inside diameter, and shall not vary from this by more than plus or minus 0.16 in. (0.4 cm). The depth of the sieve from the top of

the frame to the cloth shall not be less than 0.75 in. (1.9 cm.).

(*b*) The frames shall be constructed of first quality sheet brass in such a manner as to be permanently rigid. To prevent the material being sieved from catching in the joint between the cloth and the frame, the joint shall be smoothly filled with solder or so made that the material will not catch.

Label Marking

5. Each sieve (except the 3-in. sieve) shall bear a label marked with the following information: the designation of the sieve (the nominal size of the opening in inches for the coarse series, and the micron designation or the U. S. Standard Sieve Series Number for the fine series), the name of the series (for example, "U. S. Standard Sieve Series," "American Standard Series," or a specific manufacturer's series), the name of the manufacturer or responsible distributor, and the opening in inches and millimeters.

NOTE 4.—The requirements prescribed in Section 5 shall not be considered as requiring that the opening in inches be given twice on the labels for sieves of the Coarse Series, or that the opening in millimeters be given in addition to the micron designation on the labels for sieves of the Fine Series.

ROUND-HOLE PLATE SCREENS (SIEVES)

Plates

6. Plates used in the manufacture of round-hole screens shall be made of brass, bronze, steel, or other rigid metal. Thickness of plates shall be governed by size of openings as well as screening area of screens and shall conform to the requirements prescribed in Table II.

Type of Frame

7. Frames for laboratory screens shall be at least 8 in. in diameter. Frames for standard 8-in. laboratory screens shall conform to the requirements specified in Section 3 for woven wire-cloth sieves. Frames for large screens may be made of either hardwood or metal and may be square, rectangular, or circular, as specified. For screens having circular openings 1 in. in diameter or larger, frames larger than 8 in. in diameter are recommended.

TABLE II.—THICKNESS OF PLATES FOR ROUND-HOLE SCREENS.

Screening Area, sq. in.	Diameter of Opening, in.	Thickness of Plate, in.	
		Minimum	Maximum
Under 100	All sizes	0.049	0.066
100 and over	⅛ and ¼	0.049	0.066
	¼ to 2½, incl.	0.060	0.100
	3 and 3½	0.075	0.130
	4 and 5	0.105	0.160
	6 and 8	0.120	0.175

Spacing of Openings

8. (*a*) Spacing of openings shall conform to the following requirements:

Nominal Diameter of Opening, in.	Nominal Width of Metal Between Adjacent Openings, in.
1/16	3/64
1/8	3/32
1/4	1/8
3/8	1/16
1/2	1/16
5/8	3/64
3/4	1/16
7/8	1/16
1	3/32
1 1/4	3/32
1 1/2	1/16
2	1/16
2 1/2	3/32
3	1/16
3 1/2	1/16
4	3/64
5	1
6	1
8	1

(*b*) The openings shall be so arranged that their centers lie at the vertices of

triangles which are approximately equilateral within the limits given by the permissible variations in width of metal and diameter of opening.

Permissible Variations for Openings and Spacings

9. (*a*) For screens having openings $\frac{1}{4}$ in. or less in diameter, the actual diameter of any opening shall not vary from the nominal diameter by more than plus or minus 5 per cent.

(*b*) For screens having openings over $\frac{1}{4}$ in. in diameter, the actual diameter of any opening shall not vary from the nominal diameter by more than plus or minus 3 per cent.

(*c*) The width of metal between the adjacent openings in the screen plate shall not vary from the nominal value given in Section 8 (*a*) by more than plus or minus 20 per cent.

SQUARE-HOLE PLATE SCREENS (SIEVES)

Square-Hole Screens

10. Where square-hole plate screens are specified for use, the openings shall be the same as the openings of woven wire cloth sieves, but in other respects, except for the arrangement of the openings, they shall conform to the requirements for round-hole screens specified in Sections 6 to 9.

APPENDIX

METHOD OF CALIBRATING WOVEN WIRE CLOTH SIEVES

A1. The first test of any sieve should be to determine whether it conforms to the specifications. If a suitable standard of powdered or granular material is available for a fineness test, that test is advisable as an additional means of calibration. Pieces of unmounted sieve cloth should be tested in sections of a size suitable for mounting in the sieve frames. In some cases it may be desirable to make a detailed systematic microscopic test of the sieve, plotting the frequency of occurrence of different sizes of openings across the sieve, rejecting, if necessary, a sieve found to have an excessive nonuniformity of sizes of openings for the particular purpose for which the sieve is to be used.

A2. The diameters of the openings of the round-hole screens should be measured by means of an accurate steel rule or by other suitable means. The use of "paddle gages" (flat plug gages) or of tapered pin gages is recommended for checking these openings more precisely.

A3. To determine whether a sieve conforms to the foregoing specifications, the apparatus used may be of the general type as described below and in the National Bureau of Standards *Letter Circular 72*, July 26, 1922, and the test method may follow the procedure herein described:

The apparatus consists of a light-tight box about 40 cm. square and 1 m. in length, with a microscope mounted on one end and a ground-glass plate 2 mm. in thickness on the other end. The source of illumination is a microscope illuminator containing a concentrated filament lamp, 6 v., 108 w., connected through a transformer to a 110-v. alternating current supply circuit. The light passes through a lens in the end of the illuminator and is focused on the objective of the microscope. After passing through the microscope it diverges to the ground glass plate which is mounted with the ground side in. A 50-cm. steel scale is mounted against the inner face of the ground-glass plate in such a way that the graduations of the scale may be seen through the glass. The position of the scale allows a direct reading on the edges of the image cast by the wire of the sieve and avoids parallax due to the thickness of the glass. By oiling the ground surface slightly, the visibility is greatly increased without diminishing the distinctness of the image.

A frame for holding the sieve is placed on a platform so arranged as to permit a lateral motion of about 8 in., and also motion at right angles for focusing. Long rods, extending to the end of the apparatus at which the observer is seated, enable the observer to move the sieve without leaving his place, the lateral motion being accomplished by means of a rack and pinion and the focusing by the use of beveled gears. If the frame were also provided with a

vertical motion, the utility of the apparatus would be increased. A green glass filter is placed between the lamp and the condensing lens. The filter relieves eye strain very considerably and practically eliminates the color bands otherwise appearing on the edges of the image.

In use, the sieve is mounted in its holder on the focusing platform, between the illuminator and the objective of the microscope, and is focused by the observer until a sharp image is seen on the ground glass. Measurements are then taken in millimeters by reading the positions on the steel scale where the two edges of the image of the wire cross it, a reading glass being sometimes used. The sieve is then moved across the field, readings being taken at several places on the cloth, until the whole diameter of the sieve has been traversed, care being taken at the same time to watch for the uniformity of spacing and to measure any excessively large openings. The sieve is then rotated through 90 deg. and the process repeated.

The magnification of the apparatus may be determined by means of a calibrated stage micrometer.

By using a microscope having a tube about 15 cm. long and an eyepiece with a magnifying power of approximately eight diameters, together with a 16-mm. objective, a magnification of about 250 diameters is obtained.

The National Bureau of Standards has found by experience that in testing sieves for conformity to standard specifications, the most reliable results are obtained by measuring the wire diameters and determining the number of wires per centimeter, and then computing the average opening. From five to ten wires, sometimes more, are measured depending upon the uniformity of diameter of the wire, the closeness of the measurements of the cloth to the limits permitted by the permissible variations, and the experience of the observer. Four measurements are made on each wire. Large openings are measured at the same time.

Magnifications found suitable and convenient at the National Bureau of Standards are:

Sieves	Magnifications
590 micron (No. 30) and coarser	45
500 to 250 micron (Nos. 35 to 60), incl.	90
210 to 37 micron (Nos. 70 to 400), incl.	250

Greater magnifications would be feasible if the distance from the microscope to the ground-glass plate were increased, provided the optical parts of the microscope were of sufficient quality.

In making all measurements. the readings of the steel scale are estimated and recorded to 0.1 mm.

The most generally used method of determining the *mesh* of the sieve is by means of what is sometimes known as "picket-fence interference," also the "moire effect." For the No. 200 sieve, a glass scale is accurately graduated with 200 lines to the inch, the opaque lines being about equal in width to the space between the lines. The exact number of lines per unit length is immaterial, however, provided the graduation is uniform and the exact number per unit length is known. When a scale such as this is laid on a piece of woven wire cloth having a mesh per unit length approximating the number of lines per unit length on the scale, and a strong light is placed beneath, dark bands will appear which in a unit length are equal in number to the difference between the mesh of the cloth and the graduation of the scale. If the scale is moved length-

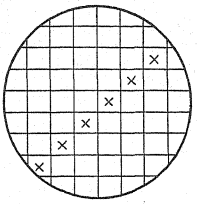


FIG. 1—Method of Selecting Openings for Plotting of Different Sizes of Openings (see Paragraph A4).

Only the indicated openings are measured.

wise, in a direction perpendicular to the lines, the dark bands will also move. If these bands move in the same direction as the motion of the scale, the mesh of the sieve is less, if the motion is in the opposite direction, the mesh is greater than the number of lines on the scale. If some bands move in one direction and some in the other, the number moving in each direction must be counted separately, and the algebraic sum taken as the number of bands. For sieves 840 micron (No. 20) to 210 micron (No. 70), inclusive, the best results are obtained with a transparent line about the width of a wire diameter ruled on an opaque background. For sieves coarser than these it is usually necessary to count the fringes by the aid of a hand lens using a steel scale as the standard. Scales should be calibrated before being used in testing sieves.

The limiting values for average opening and for maximum opening are found by multiplying

the nominal values in column 2 of Table I by the permissible variations expressed in per cent (columns 4 and 5 of Table I)—considered as exact figures followed by zeros after the decimal point—and rounding off the result to the same number of decimal places as given in column 2 of Table I.

A4. If it is desired to plot the frequency of occurrence of different sizes of openings across the sieve, 100 openings in the sieve should be measured in a diagonal direction across the sieve, then 100 openings are measured in a diagonal direction at right angles to the first. Six fields are chosen in each direction, and in any one field the diagonal method of measurement is used as illustrated in Fig. 1

Each opening is measured between the warp wires and also between the shoot wires; the warp readings and the shoot readings are separately tabulated and plotted.

A5. The National Bureau of Standards accepts sieves for test to determine conformity to specifications.

A6. Glass scales such as are described above may be obtained from: Bausch & Lomb Optical Co., Rochester, N. Y., and Keuffel & Esser Co., Hoboken, N. J.

PRESENTATION OF DATA

A7. Sieve tests should be presented in tabular or graphical form in terms of the nominal sieve opening and the percentage by weight. For purposes of comparison the cumulative percentage undersize will be accepted as standard. This does not preclude the representation of percentages on individual sieves, provided the sieve interval is clearly specified as plus one sieve number and minus another, or as between two sieve apertures. Graphical representation may include the use of logarithmic scales, probability paper, etc., to emphasize specific characteristics of shape.

A8. The presentation of data for round-hole screens should follow the same general procedure as that specified in Paragraph A7 of this Appendix for square apertures, but in all cases the term *diameter* should be applied to the size of the opening.

AMERICAN ASSOCIATION STATE
HIGHWAY OFFICIALS STANDARD
A.A.S.H.O. NO.: T 67-42

Standard Methods of
VERIFICATION OF TESTING MACHINES[1]

A.S.T.M. Designation: E 4 - 36

ADOPTED, 1936.[2]

Reapproved in 1942 Without Change.

This Standard of the American Society for Testing Materials is issued under the fixed designation E 4; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods cover procedures for the verification of testing machines by means of standard calibrating devices.

DEFINITIONS

Definitions

2. (*a*) *Testing Machine.*—A mechanical device for applying a load (force) to a specimen.

NOTE.—Usually the magnitude of the load can be changed at the will of the operator. Many testing machines are arranged to measure the load, but this is not always the case, especially with impact machines and machines for testing ductility.

(*b*) *Accurate.*—A testing machine is said to be accurate if the indicated load is within the specified permissible variation from the actual load.

NOTE.—The word "accurate" applied to a testing machine shall be used without numerical values, for example, "An accurate testing machine was used for the investigation."

The *accuracy* of a testing machine should not be confused with *sensitiveness*. For example, a testing machine might be very sensitive, that is, it might indicate quickly and definitely small changes in the load, but, nevertheless, be very inaccurate. On the other hand, the accuracy of the results is, in general, limited by the sensitivity.

(*c*) *Error.*—In the case of a testing machine, the value obtained by subtracting the correct value of the quantity measured (usually load) from the indicated value as given by the testing machine.

NOTE.—The error may be positive or negative.

The word "error" shall be used with numerical values, for example, "At a load of 30,000 lb. the error of the testing machine was 15 lb."

(*d*) *Percentage of Error.*—In the case of a testing machine, the ratio, expressed as a percentage, of the error to the correct value of the quantity measured.

(*e*) *Correction.*—In the case of a testing machine, the value obtained by subtracting the indicated value from the correct value of the quantity measured.

NOTE.—The correction has the same magnitude as the error but the opposite sign. It is recommended that, except for special cases, no

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee E-1 on Methods of Testing.

[2] Prior to their present adoption as standard, these methods were published as tentative from 1923 to 1924, being revised in 1924. They were adopted in 1924, but withdrawn and republished as tentative from 1933 to 1936, being revised in 1934, 1935, and 1936. Editorially revised and rearranged in 1939.

corrections be used on machines tested and found to have errors within the permissible variations given in these methods.

(*f*) *Permissible Variation.*—In the case of a testing machine, the maximum allowable error in the value of the quantity indicated.

NOTE.—It is convenient to express permissible variation in terms of percentage of error. The numerical value of the permissible variation for a testing machine is so stated hereafter in these methods.

(*g*) *Loading Range.*—The loading range of a testing machine or of an apparatus for calibrating a testing machine is the range of indicated loads for which the testing machine or the calibrating apparatus gives results within permissible variations specified.

(*h*) *Elastic Calibration Device.*—An elastic calibration device for use in verifying the load readings of a testing machine consists of an elastic member to which loads may be applied combined with a mechanism for indicating the magnitude of deformation under load.

METHODS FOR VERIFYING TESTING MACHINES THAT MEASURE LOAD

Advantages and Limitations of Different Methods.

3. Four methods of verifying testing machines are listed in this standard, as follows:

(*a*) *Verification by Standard Weights.*—Verification by the direct application of standard weights to the weighing mechanism of the testing machine, where practicable, is the most accurate method. Its limitations are (*1*) the small range of load which can be covered, (*2*) the nonportability of any large amount of standard weights, and (*3*) its nonapplicability to horizontal testing machines or to vertical testing machines whose weighing mechanisms are attached to their upper platens.

(*b*) *Verification by Proving Levers.*—Verification by the use of standardized proving levers loaded with standard weights ranks second in accuracy. Its limitations are (*1*) the fact that the range of load possible with proving levers, while greater than with standard weights, is not great enough to cover the capacity range of large testing machines, and (*2*) the inconvenience of transportation of proving levers and standard weights for verifying large testing machines.

(*c*) *Verification by Elastic Calibration Device.*—Verification by the use of an elastic calibration device ranks third in accuracy. It is free from the limitations of the methods referred to in Paragraphs (*a*) and (*b*).

(*d*) *Verification by Comparison Method.*—Verification by a comparison of the tensile strength of test specimens ("companion specimens") cut from the same piece of metal, is distinctly less accurate than the three methods referred to in Paragraphs (*a*), (*b*), and (*c*), and should be used only when none of the other methods are available. If the results of a verification test by the comparison method fail to agree with the results of a verification test by any of the other three methods, the results given by the comparison method shall be discarded.

Method of Applying Load

4. In the verification of a testing machine, the loads shall be applied in ascending order.

NOTE.—For any testing machine (particularly for certain types of machines in which the load-indicating device is actuated by a Bourdon pressure tube, a hydraulic (or steam-engine) indicator, or other device depending on the elastic properties of a material, or for machines in which the load is measured by measuring the pressure in a hydraulic jack), the errors observed at corresponding loads taken first by increasing the load to the test load and then by decreasing it to the test load may not agree. Testing machines are usually used under in-

creasing loads, but if a testing machine is to be used under decreasing loads it should be calibrated under decreasing loads as well as under increasing loads.

Selection of Test Loads

5. For any loading range, the testing machine shall be verified by at least five test loads (except for testing machines designed to measure only a smaller number of definite loads, such as certain hardness testing machines). The difference between any two successive test loads shall not exceed one-third of the difference between the maximum and minimum test loads.

NOTE.—A testing machine may have more than one loading range. For instance, a 100,000-lb. capacity testing machine may have one loading range from 5000 lb. to 40,000 lb. and a second loading range from 55,000 lb. to 100,000 lb. Section 4 requires that the machine must be verified by at least five test loads between 5000 lb. and 40,000 lb. and also that the machine must be verified by at least five test loads between 55,000 lb. and 100,000 lb.

Eccentric Loading

6. For the purpose of determining the loading range of a testing machine, all calibration loads shall be applied so that the resultant load shall be as nearly along the axis of the testing machine as is possible.

NOTE.—The effect of eccentric load on the accuracy of a testing machine may be determined by calibration readings taken with proving levers or an elastic calibration device placed so that the resultant load is applied at definite distances from the axis of the machine, and the loading range determined for a series of eccentricities. In the case of testing machines in which the load reading depends on the hydrostatic pressure in a cylinder fitted with a piston, the effect of eccentricity of loading is most serious when the piston is at the extreme outward position allowable.

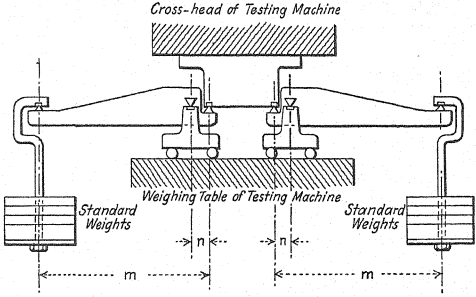


FIG. 1.—Proving Levers.

Verification by Standard Weights

Procedure

7. Standard metal weights of suitable design, finish, and adjustment shall be placed on the weighing platform of the testing machine or upon trays or other supports suspended from the load-measuring mechanism in place of the specimen. The weights shall be applied in increments and removed in the reverse order. They shall be arranged symmetrically with respect to the weighing platform, so that the center of gravity of the load lies in the vertical line through the center of the platform.

The applied load and the indicated load shall be recorded for each test load applied, and the error calculated from these data.

NOTE.—The method of verification by standard weights can be used only on vertical testing machines. The total load is limited by the size of the platform and the number of weights available. Often 20 weights of 50 lb. each are used, a total of 1000 lb.

This method of verification is seldom used for any but small testing machines, as the loading range does not include the loads for which large machines are used.

Verification by Standardized Proving Levers

Levers

8. (*a*) The arrangement of proving levers for verifying testing machines is shown in Fig. 1. The two levers rest on supports on the weighing platform of the testing machine. These supports shall move easily in a horizontal direction, which insures that the forces at each of the knife edges shall be very nearly vertical. The inner knife edges in each lever bear against a suitable block in the movable head of the testing machine. Weight trays or hangers are suspended from each of the outer knife edges and these trays or hangers are loaded with standard weights. The increment of load put on the testing machine by the standard weights is the amount of standard weights multiplied by the lever ratio m/n, Fig. 1.

(*b*) The knife edges, as well as their supports, shall be of hardened tool steel. The knife edges shall be ground sharp to an angle of 90 deg. The load on any knife edge shall not exceed 7000 lb. per linear inch. The three knife edges in each lever shall be parallel, and their edges shall lie in a plane. Each lever shall have machined surfaces, in this or in some parallel plane, upon which a spirit level can be placed.

Verification of Levers

9. The lever ratio of a proving lever shall be determined by the use of at least three test loads; the amount of weights used shall not be less than the maximum load applied upon one of the arms of a lever in using the levers to verify testing machines. The proving lever shall be balanced over its center knife edge with suitable weight trays suspended from the end knife edges. Standard weights shall then be applied to the trays in three steps, corresponding approximately to 50, 75, and 100 per cent of the weights available, and the proving lever shall then be brought to a balance by the use of small weights and by observations of the freely swinging proving lever. From the weights in the two weight trays the lever ratio shall be calculated.

Procedure

10. The proving levers shall be placed symmetrically in the testing machine to be verified, and both levers shall be brought as near to a horizontal position as is feasible, after applying each increment of load, by means of the movable head of the testing machine. The testing machine shall be balanced with the levers in place and the weight trays empty. Standard weights shall be applied (or removed) in increments, half an increment in (or from) each tray. The weights shall be placed symmetrically on the weight trays, with the center of gravity of the weights over the center of the tray. The applied load and the indicated load shall be recorded for each test load applied, and the error calculated from these data.

NOTE.—The use of standardized proving levers on horizontal testing machines involves the use of bell crank levers. They require special methods of determination of lever ratio, which have not yet been codified in A.S.T.M. standards. Proving levers for vertical testing machines are now available up to 50,000-lb. capacity.

Verification by Means of an Elastic Calibration Device[3]

Design and Workmanship

11. An elastic calibration device should be so designed and constructed that its accuracy is not in danger of being impaired by handling, shipping, or ordinary use, and so that parts subject to damage, replacement, or removal for storage and shipment can be replaced without impairing the accuracy of the device.

NOTE.—This section is nonquantitative in its requirements. It is placed in the method for the purpose of calling attention to the fact that an elastic calibration device, if improperly designed and constructed, may, like any other delicate measuring instrument, be damaged and rendered inaccurate by handling and shipping.

Shackles and Bearing Blocks

12. An elastic calibration device shall be provided with shackles, bearing blocks, or any other necessary fixtures through which the load is to be applied to the calibration device. All such fixtures should be so designed and constructed that when placed in a testing machine in reasonably good condition there shall be no variation of reading in excess of permissible variations, due to imperfections in the bearing blocks or in the motion of the head of the testing machine.

NOTE.—This section is nonquantitative in its requirements. It is placed in the method for the purpose of calling attention to the fact that poorly designed loading fixtures can render inaccurate the readings of an elastic calibration device.

Graduation of Scale

13. (*a*) The scale of the indicating mechanism shall be uniformly graduated. The distance between any two graduation lines, dots, or other dividing marks (or between their images, if a viewing system with a definite optical magnification is used), shall be not less than 0.035 in.

(*b*) The difference between the position of any graduation line and its correct position on the scale shall not exceed one twentieth of the distance between two adjacent graduation lines. For scales on a circular dial, when successive lines are set to one fixed index line, the positions of successive graduation lines nearly diametrically opposite, referred to another fixed index line shall differ from each other by (*1*) not more than 5 per cent of the smallest division of the dial, or (*2*) not more than 0.1 per cent of the minimum load in the loading range of the instrument, if (*2*) be larger than (*1*).

(*c*) The change of load necessary to cause a relative movement of 0.1 in. between the index line and the scale (or its image if a viewing system with a definite optical magnification is used) shall not exceed 1 per cent of the capacity load of the instrument.

Label Showing Manufacturer, Number, and Capacity

14. The manufacturer's name, the capacity load, and the serial number of the device shall be legibly marked on some part of the instrument.

Temperature Equalization

15. When using an elastic calibration device to verify the load readings of a testing machine, the device should be placed near, or preferably in, the testing machine a sufficient length of time before the test so that the device and the testing machine shall be at very nearly the same temperature.

[3] The term "calibration device" shall be interpreted to include solid bars, hollow bars, elastic loops, and other members whose elastic deformation can be measured.

Elimination of Small Temperature Effects Due to Stress

16. To minimize errors due to temperature effect of stress and pseudo-elastic action, the time schedule followed when verifying an elastic device which is loaded in axial tension or compression shall be reported; and the same time schedule, within limits stated in the report, shall be followed when using the device to calibrate a testing machine.

Primary Standards for Elastic Calibration Devices

17. (*a*) For loads not exceeding 100,000 lb., an elastic calibration device shall itself be calibrated by applying dead weights known to be accurate within 0.02 per cent.

(*b*) For loads exceeding 100,000 lb., an elastic calibration device may be calibrated by another elastic calibration device or by a combination of several elastic calibration devices, or by proving levers[4] and dead weights. In this case the calibrating load applied shall be known to be accurate within 0.1 per cent.

Preliminary Cyclic Loading

18. Before taking readings for the formal verification of an elastic calibration device by means of dead weights or other primary standards, the device shall be subjected to a series of cyclic loads varying from a minimum load not greater than 2 per cent of the capacity load of the device to a maximum load not less than 5 per cent nor more than 10 per cent above the nominal capacity load.

Routine in Calibration of Elastic Calibration Device

19. For any range of test loading which may be applied in calibrating an elastic calibration device, there shall be applied at least eight test loads, and the difference between any two successive test loads shall not exceed one-fifth of the difference between the minimum and the maximum test loads. The series of test loads shall be applied to the elastic calibration device at least three times, and the shackles or bearing blocks shall be readjusted before each application of a series of loads.

NOTE.—If the magnitude of deformation of the elastic calibration device is measured with a self-indicating dial micrometer or dial indicator, the elastic calibration device shall be used only at the test loads for which it has itself been calibrated by comparison with a primary standard. It shall be used only when the deformation measuring apparatus is so adjusted that the no-load reading of the device is nearly equal to the no-load reading observed during the calibration of the elastic calibration device.

Permissible Variations for Elastic Calibration Device

20. (*a*) For ordinary use, the loading range of an elastic calibration device shall be that range of load within which any reading under an applied load shall not differ from the average of at least three readings under the same load by more than one-fifth of 1 per cent.[5]

(*b*) The report of the verification of the elastic calibration device shall also state the loading range within which no reading varies from the average of at least three readings under the same load by more than one-tenth of 1 per cent. This range shall be designated as the *Precision Loading Range* for the device.

Temperature Correction for Elastic Calibration Device

21. In using an elastic calibration device made of steel with not more than 5 per cent of alloying elements, a fairly accurate correction for variation in temperature may be made on the basis

[4] The term "proving levers" is not to be interpreted as allowing the use of the lever system of a compound lever testing machine or of a platform scale as a part of a primary standard.

[5] This means that the report of a standardizing laboratory on an elastic calibration device will state *within what loading range* it may be used, rather than reporting a blanket acceptance or rejection of the device. This follows the practice recommended for testing machines.

that the modulus of elasticity in tension or compression (or flexure) diminishes by 0.015 per cent for each degree Fahrenheit increase in temperature. For other metals, if the change in the modulus of elasticity with temperature is known, a correction for temperature should be applied to the readings of the instrument. During any calibration of a testing machine with an elastic calibration device, or during the calibration of the device itself, temperature shall be kept as uniform as possible.[6]

Verification by Companion Specimen Method

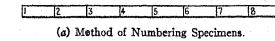
Description of Comparison Method

22. When the direct method of verification of a testing machine cannot be carried out (as is the case at present with horizontal testing machines, for example), or when an elastic calibration device is not available, a comparison method of verification may be used. In this method the machine to be verified is tested by comparing its indications with the corresponding readings obtained under the same conditions by the use of another testing machine, especially tested and verified, as specified in Section 23. The method of verification by comparison shall be carried out by the use of a series of companion specimens, half of which are to be tested in tension in the machine to be verified, and half of which are to be tested in tension in the especially verified testing machine which serves as a standard machine. The general provisions respecting the application of the test loads and the loading range which are outlined in the direct method of verifying testing machines (Sections 4, 5, and 6) shall apply to the verification of testing machines by the comparison method, so far as is practicable.

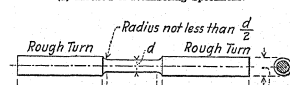
Requirements for Testing Machines Used for Verifying Other Machines

23. (*a*) A testing machine which is to be used as the standard for verifying other testing machines shall itself be verified by a special test, in which the verification is carried out twice. The loading range shall be determined by the permissible variation of plus or minus 1 per cent (Section 27). For the loading range established, the differences in indications found in the two tests of the standard machine shall not exceed 0.25 per cent.

(*b*) In verifying testing machines by the companion specimen method, corrections shall be applied to the indications of the standard machine and these corrections shall be based on the average



(*a*) Method of Numbering Specimens.



(*b*) Form of Test Specimen.

FIG. 2.—Companion Specimen.

correction obtained in the two tests used in its verification.

Test Specimens

24. (*a*) For each test load, eight or more tension test specimens shall be cut from soft-rolled or drawn steel and numbered consecutively, as in Fig. 2 (*a*).

(*b*) The tensile strength of the steel shall be determined by a preliminary test and the sets of specimens for the comparison test shall have such nominal cross-sectional areas as will give, approximately, the loads required. The form of the specimen shall be as shown in Fig. 2 (*b*). The ends may be threaded or otherwise machined to fit holders,

[6] Similar errors due to change of modulus of elasticity with temperature may be present in the load-indicating mechanism of a testing machine using a Bourdon gage or other device dependent on the elastic deformation of a member.

but it is recommended that those specimens shipped to another laboratory be left with cylindrical ends. The actual value of "*d*" (Fig. 2 (*b*)) for each specimen shall be determined by means of a micrometer.

NOTE.—It should be noted that the standard tension test specimen, 0.5 in. in diameter and having a 2-in. gage length, satisfies the requirements for a specimen for the verification of a testing machine by the comparison method. See Fig. 3 of the Standard Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8) of the American Society for Testing Materials.[7]

Procedure

25. (*a*) The specimens having odd numbers shall be tested in the machine to be verified, and those having even numbers shall be tested in the standard testing machine. The tensile strength only shall be determined.

(*b*) The speed of the testing machine, by which is meant the speed of the pulling head when the machine is running idle with no specimen in the machine, when testing verification specimens shall be approximately the same for both the standard testing machine and the machine being verified. The speed shall be the slowest speed that can be obtained on both machines; in any case the speed shall not exceed 1 in. per min. per inch of diameter of companion specimen.

(*c*) When testing companion specimens, especial care shall be taken to maintain the weighing beam of the testing machine in equilibrium as the tensile strength of the specimen is approached. It is recommended that a mark be placed at the middle of the trig loop, and that a pointer be placed on the beam for verification tests, and that the pointer be kept as nearly on the mark as possible.

[7] 1944 Book of A.S.T.M. Standards, Part I.

Calculation of Error

26. (*a*) The average tensile strength (in pounds per square inch) of the odd-numbered specimens, multiplied by the nominal area of cross-section, shall be considered a measure of the indicated load, and the average tensile strength of the even-numbered specimens, multiplied by the nominal area of cross-section, shall be considered a measure of the applied load. The error shall be calculated from these data.

(*b*) If the tensile strength (in pounds per square inch) of any companion specimen varies more than 1 per cent from the mean of the group with which it is tested, its tensile strength shall not be used in calculating the error of the testing machine. If more than one specimen in the odd-numbered group or more than one specimen in the even-numbered group exceeds the permissible variation in tensile strength, all the specimens shall be discarded and another set prepared.

PERMISSIBLE VARIATIONS FOR TESTING MACHINES THAT MEASURE LOAD

Permissible Variation Within Loading Range of a Testing Machine

27. The error for loads within the loading range of a testing machine shall not exceed 1 per cent (Notes 1 and 2).

NOTE 1.—This means that the report of the verification of a testing machine will state *within what loading range it may be used*, rather than reporting a blanket acceptance or rejection of the machine.

NOTE 2.—In no case shall the loading range be stated as including loads below a value which is 100 times the smallest load which can be read on the testing machine. (This means that if a testing machine had graduations so spaced that it can be read to within $\frac{1}{5}$ division and the machine is sensitive to $\frac{1}{10}$ division it would be possible for the loading range to extend down to the load corresponding to 10 divisions. On the other hand, if the testing machine was sensitive to only 2 divisions, the

loading range could not extend below the load corresponding to 200 divisions.)

Corrections

28. The indicated load of a testing machine shall not be corrected either by calculation or by the use of a calibration diagram to obtain values within the required permissible variation.

TIME INTERVAL BETWEEN VERIFICATIONS

Time Interval Between Verifications

29. It is recommended that testing machines, when in constant use, be verified at intervals of 6 months and, when used intermittently, at intervals of 2 or 3 yr. Testing machines shall, however, be verified immediately after making repairs or adjustments of the weighing mechanism, after the testing machine has been moved (this does not apply to portable testing machines), and whenever there is reason to doubt the accuracy of the results, without regard to the time interval since the last verification.

REPORTS AND CERTIFICATES

Reports

30. A clear and complete report shall be prepared of each verification of a testing machine. This report shall state the method of verification used, and shall give the serial numbers and the names of manufacturers of all apparatus used in carrying out the verification. It shall state how, by whom, and when the calibration of the apparatus used in verifying the testing machine was made, the loading range, the "precision" loading range of the calibration apparatus, and the loading range of the testing machine.

Certificates

31. A certificate giving the manufacturer's serial number and a brief description of the testing machine, the manufacturer's name, the date of verification, and the loading range shall be signed by the person responsible for the maintenance of the testing machine, and this certificate shall be posted in plain view of a person operating the testing machine.

Standard Definitions of
TERMS RELATING TO METHODS OF TESTING[1]

A.S.T.M. Designation: E 6 – 36

ADOPTED, 1930; REVISED, 1936.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation E 6; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Stress.—The intensity (measured per unit area) of the internal distributed forces or components of force which resist a change in the form of a body. Stress is measured in force per unit area (pounds per square inch, kilograms per square millimeter, etc.).

NOTE.—In examining the definitions given in various authoritative textbooks two definitions of the term "stress" were found. Textbooks in physics and some European textbooks in mechanics of materials gave a definition substantially as above. American textbooks in engineering mechanics define stress as a *force*, measured in *pounds*. They call the intensity of force (pounds per square inch) "unit stress," "intensity of stress," or "fiber stress." This use of the term stress is illustrated by the "stress sheet" of the bridge engineer which gives *forces*, measured in pounds.

In view of the fact that even in engineering textbooks the term "stress" is often used to denote intensity of force per unit of area, and in view of the fact that the physicists' definitions involve somewhat simpler terms, the physicists' definitions have been followed in these standards.

While it is important to have a clear definition for the term "stress" it is even more important to keep clearly in mind the *units* used in calculations and test data for materials of construction. In fact, if the units are always given, there can be no misunderstanding as to the sense in which the term "stress" is used.

There are three kinds of stress: tensile, compressive, and shearing. Flexure involves the combination of tensile stress and compressive stress. Torsion involves shearing stress.

It is customary to calculate stress on the basis of the original dimensions of the cross-section of the body.

Strain.—The change per unit of length in a linear dimension of a body, which change accompanies a stress. Strain is measured in inches per inch of length (millimeters per millimeter).

NOTE.—In some American engineering textbooks the term "strain" is used in the sense of total deformation and is measured in inches. Change of dimension per unit length is called "unit strain," or "unit deformation." As in the consideration of the term "stress," the definitions given in textbooks in physics have been followed.

Under tensile stress or compressive stress, strain is measured along the dimension under consideration. Shearing strain is measured at right angles to the dimension under consideration. In torsion tests, which involve shearing stress, it is customary to measure

[1] Under the standardization procedure of the Society, these definitions are under the jurisdiction of the A.S.T.M. Committee E-1 on Methods of Testing.

[2] Prior to adoption as standard, these definitions were published as tentative from 1923 to 1930, being revised in 1924 and 1925.

the angle of twist, which may be translated into terms of strain.

Stress-Strain Diagram.—A diagram plotted with values of stress as ordinates and values of strain as abscissas.

NOTE.—The use of the term stress-strain diagram is frequently extended to cover diagrams plotted with values of applied load, or applied moment as ordinates, and with values of stretch, compression, deflection, or twist as abscissas.

Fig. 1 is an example of a stress-strain diagram.

Stress-strain diagrams are in some cases drawn directly by an autographic attachment to the testing machine. A more usual method of procedure consists in taking a series of load readings (from the balanced scale beam of the testing machine) with corresponding readings of the strain-indicating apparatus (extensometer, compressometer, deflectometer, or torsion indicator). The term "strainometer reading" will be used to denote the reading of any strain-measuring instrument. From these readings, or from values calculated from them, there is plotted a diagram with stress-indicating values (load, flexural moment, twisting moment, or stress) as ordinates, and strain-indicating values (elongation, shortening, deflection, twist, or strain) as abscissas. In planning such a test it is necessary to decide on the increment of load or the increment of reading of strainometer to be used between successive readings.

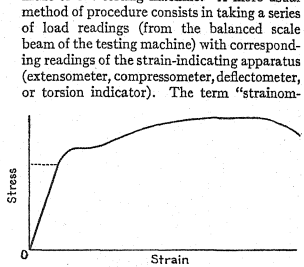


FIG. 1.—A Typical Stress-Strain Diagram with Elastic Strains Exaggerated.

In Fig. 2 are shown three typical stress-strain diagrams. The material for all three tests is the same, and the test specimens are all of the same size, so that the diagrams should be the same. The diagram shown in Fig. 2 (*a*) is plotted from points determined by taking increments of load (*S*). In this diagram it is seen that owing to the shape of the curve, data for locating points between *M* and *N* are lacking. That particular portion of the curve (the "knee" of the curve) is the part for which it is especially desirable to locate several points on the diagram.

In Fig. 2 (*b*) is shown a diagram plotted from points determined by taking increments of strainometer reading (*e*). It will be noted that for this diagram there are located several points near the knee of the curve, and the shape of the diagram in this important region is much more definitely determined than for the curve shown in Fig. 2 (*a*).

The custom of choosing increments of load rather than increments of strainometer reading is quite common in tests of materials and is followed because, in general, it is easier to calculate increments of load than it is to calculate increments of strainometer reading. An estimate of the load necessary to stress the specimen up to the knee of the curve is made and some fraction (frequently one-tenth) of this value is taken as an increment. The calculation of the corresponding increment of strainometer reading is more complicated.

Sometimes the practice is followed of applying a few increments of load as determined above, and then applying load in much smaller increments (*S'*) until the knee of the curve is passed. Fig. 2 (*c*) shows a diagram obtained in this manner. This method involves a marked increase in the number of readings necessary for a test and with unknown material there is always some danger that the knee of the curve will be reached before the use of small increments of load is begun.

The following method of choosing increments for a test is suggested: Estimate the load corresponding to the knee of the stress-strain diagram and choose a value for increment of load about one-tenth of this value. Apply this increment of load (*S*) once and note the corresponding change of reading for the strainometer (*e*). Then for the remainder of the test, use for the increment of strainometer reading a value which corresponds to some convenient interval on the scale of the strainometer, and which is approximately equal to (*e*).

Elastic Limit.—The greatest stress which a material is capable of developing without a permanent deformation remaining upon complete release of the stress.

NOTE.—It is a matter of experience with many materials, especially with many metallic materials, that using ordinary methods of testing, the values found for elastic limit by means of observations of permanent deformation (set) after release of stress do not differ

widely from the values found for proportional limit, the latter being defined as:

Proportional Limit.—The greatest stress which a material is capable of developing without a deviation from the law of proportionality of stress to strain (Hooke's Law).

Since the determination of proportional limit is much more readily made than is the determination of elastic limit, it is customary to accept the proportional limit as equivalent to the elastic limit for such materials, and hence the proportional limit is frequently called the "proportional elastic limit."

For certain grades of steel a special method of determining elastic strength is recognized in the standard specifications of the American Society for Testing Materials. See the Standard Specifications for Carbon-Steel Forgings for General Industrial Use (A.S.T.M. Designation: A 235),[3] for Carbon-Steel Forgings for Locomotives and Cars (A.S.T.M. Designation: A 236),[3] for Alloy-Steel Forgings for General Industrial Use (A.S.T.M. Designation: A 237),[3] and for Alloy-Steel Forgings for Locomotives and Cars (A.S.T.M. Designation: A 238).[3]

Yield Strength.—The stress at which a material exhibits a specified limiting permanent set.

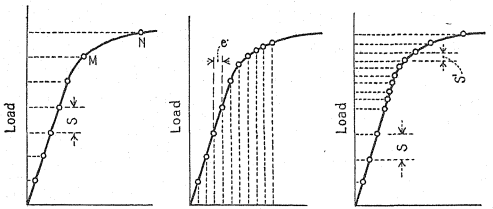
NOTE.—It is usually impracticable and probably impossible to determine the stress at which inelastic action in a member *begins*. Plastic yielding in nearly all members (including the specimen in a carefully controlled laboratory test) starts as local actions and becomes measurable only after many local internal adjustments and accommodations have occurred, and after a considerable portion of the member is affected by the yielding.

The limit of usefulness of many materials, especially metals, in members subjected to approximately static loading at ordinary temperatures is therefore determined by a measurable value of plastic yielding of the material above which the material is considered to be damaged and below which the damaging effects are considered to be negligible.

The following methods are recommended for determining the yield strength of a material:

1. For material that has a "sharp-kneed" stress-strain diagram and hence exhibits at a certain stress the special characteristic of yielding without increase in stress. Two satisfactory methods are in use:

(*a*) *"Drop of the Beam" Method.*—In this method the load is applied to the specimen at a steady rate of increase and the operator keeps the beam in balance by running out the poise at approximately a steady rate. When the yield strength of the material is reached the increase of load stops, but the operator runs the poise a trifle beyond the balance position and the beam of the machine drops for a brief but appreciable interval of time. In a machine fitted with a self-indicating load-measuring device, there is a sudden halt of the load-indicating pointer corresponding to the drop of the beam. The load at the "halt" or the



(*a*) With Equal Increments of Stress. (*b*) With Equal Increments of Strain. (*c*) With Two Values of Increments of Stress.

FIG. 2.—Illustrating Three Methods of Plotting Load-Deformation Curves.

[3] 1944 Book of A.S.T.M. Standards, Part I.

"drop" is recorded, and the corresponding stress is taken as the yield strength. This method of determining the yield strength requires only one man to conduct a test.

(*b*) *Total Strain Method Using Dividers* (Frequently called the "*Dividers Method*").—In this method an observer with a pair of dividers or other suitable apparatus, watches for visible elongation between two section marks on the specimen. When visible stretch is observed, the load at that instant is noted, and the stress corresponding to the load is taken as the

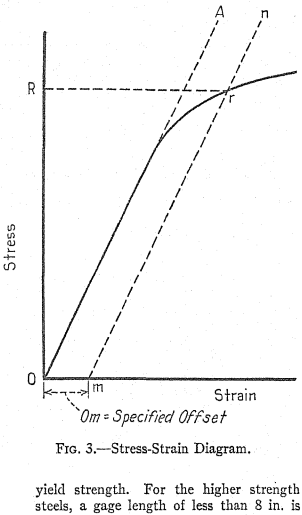


FIG. 3.—Stress-Strain Diagram.

yield strength. For the higher strength steels, a gage length of less than 8 in. is recommended.

Note on Yield Point.—The above two methods determine what is usually called the yield point which is commonly defined as follows:

Yield Point.—The stress in a material at which there occurs a marked increase in strain without an increase in stress.

It should be noted that only materials that exhibit this unique phenomenon of yielding have a yield point. The term yield point should not be used in connection with material whose stress-strain curve in the region of yield is a smooth curve of gradual curvature.

2. For material whose stress-strain diagram in the region of yield is a smooth curve of gradual curvature:

(*a*) *Offset Method.*—This method can be used, if desired, for materials having "sharp-kneed" stress-strain diagrams, but is especially adapted to materials whose stress-strain diagram in the yield range is a smooth curve of gradual curvature.

For nearly all materials, if at any point on the stress-strain diagram such as *r* in Fig. 3, the load is released, the diagram for decreasing load will follow a line *rm* approximately parallel to the initial portion *OA*, of the diagram for increasing load. The offset *Om* will then give the approximate value of the permanent set after the release of the stress *OR*. The value of this set is given in percentage of original gage length. Thus to determine the yield strength by the "offset method," it is necessary to secure data (autographic or numerical) from which a stress-strain diagram may be drawn. Then with the stress-strain diagram (Fig. 3) lay off *Om* equal to the specified value of the set, and draw *mn* parallel to *OA* and thus locate *r*, the intersection of *mn* with the stress-strain diagram. Draw *Rr* parallel to the *X* axis and then *OR* gives the value of the yield strength.

In reporting values of yield strength obtained by this method, the specified value of "offset" used should be stated in parentheses after the term yield strength. Thus:

Yield strength (offset = 0.1 per cent) = 52,000 psi. indicates that at a stress of 52,000 psi. the approximate permanent set of the material reached the value of 0.1 per cent of the original gage length.[4]

[4] Yield Strength (or its equivalent) is specified as follows in the standards and tentative standards of the American Society for Testing Materials:

Standards or Tentative Standards for: Alloy-Steel Pipe; Aluminum-Alloy Bars, Rods, Shapes, Sheet; Magnesium-Base Alloy Forgings, Castings, Sheet	0.2 per cent "offset"
The Alloy: Copper, 80 per cent; Tin, 10 per cent; Lead, 10 per cent; Copper-Silicon Alloy Plates, Sheets, and Bars; Malleable Iron Castings; Aluminum-Bronze Castings; Bronze Bearing Metals and Steam or Valve Sand Castings; Composition Brass or Ounce Metal Sand Castings; Naval Brass Rods for Structural Purposes	0.5 per cent elongation under load (approximately equivalent to 0.35 per cent "offset")

In using this method, an extensometer reading to 0.0001 in. per in. of gage length would be sufficiently sensitive for most materials. The elastic limit and the proportional limit may be regarded as special values of the yield strength. They are the highest stresses for which the "set" and the "offset," respectively, are not measurable with the instruments used and hence is considered to be zero.

For any given stress the deviation (offset) from Hooke's law is only approximately equal to the set after the removal of that stress, and the approximation becomes less and less exact as the permissible deviation diminishes. This method is devised for determining a stress corresponding to a well marked plastic deformation, or set, and it is not feasible to specify a very small value for the permissible "offset" *Om*.

(*b*) *Approximate Method Without Stress-Strain Diagram*.—For tests to determine the acceptance or rejection of material whose stress-strain characteristics are well known from previous tests of similar material in which stress-strain diagrams were plotted, the total strain corresponding to the stress at which the specified permanent set occurs will be known within satisfactory limits; therefore, in such tests a specified total strain may be used, and the stress on the specimen, when this total strain is reached, is the value of the yield strength. The total strain can be obtained satisfactorily by use of an extensometer reading to 0.0001 in. per in. of gage length. But is recommended that this approximate method be used only after agreement between the manufacturer and the purchaser, with the understanding that check tests be made for obtaining stress-strain diagrams for use with the offset method to settle any misunderstandings.

Tensile Strength.—The maximum tensile stress which a material is capable of developing.

NOTE.—In practice, it is considered to be the maximum stress developed by a specimen representing the material in a tension test carried to rupture, under definite prescribed conditions. Tensile strength is calculated from the maximum load carried during a tension test and the original cross-sectional area of the specimen.

Compressive Strength.—The maximum compressive stress which a material is capable of developing.

NOTE.—In the case of a material which fails in compression by a shattering fracture the compressive strength has a very definite value. In the case of materials which do not fail in compression by a shattering fracture the value obtained for compressive strength is an arbitrary value depending upon the degree of distortion which is regarded as indicating complete failure of the material.

Modulus of Elasticity.—The ratio, within the elastic limit of a material, of stress to corresponding strain.

NOTE.—As there are three kinds of stress, so are there three moduli of elasticity for any material: the modulus in tension, the modulus in compression, and the modulus in shear. The value of the modulus of elasticity in tension is nearly the same, for most metals, as the value of the modulus of elasticity in compression. The value of the modulus of elasticity in shear is smaller than the value of the modulus of elasticity in tension. The modulus of elasticity is expressed in pounds per square inch (kilograms per square millimeter).

AMERICAN ASSOCIATION STATE
HIGHWAY OFFICIALS STANDARD
A.A.S.H.O. NO.: M 132-42

Standard Definitions of
TERMS RELATING TO SPECIFIC GRAVITY[1]

A.S.T.M. Designation: E 12 - 27

ADOPTED, 1927.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation E 12; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Absolute Specific Gravity (of solids and liquids).—The ratio of the weight referred to vacuum of a given volume of the material at a stated temperature to the weight referred to vacuum of an equal volume of gas-free distilled water[3] at a stated temperature. It shall be stated thus:

(*a*) When the temperatures of the material and of the water are the *same:*

Absolute Specific Gravity $x°/x°$C. .....

where x is the temperature of the material and the water.

(*b*) When the temperatures of the material and of the water are *not the same:*

Absolute Specific Gravity $x°/y°$C. .....

where x is the temperature of the material and y is the temperature of the water.

NOTE.—In the interest of standardization and simplification, the first form of statement of specific gravity (*a*) should be employed wherever practicable.

Specific Gravity (of solids and liquids).—The ratio of the weight in air of a given volume of the material at a stated temperature to the weight in air of an equal volume of distilled water at a stated temperature. It shall be stated thus:

(*a*) When the temperatures of the material and of the water are the *same:*

Specific Gravity $x°/x°$C. .....

where x is the temperature of the material and the water.

(*b*) When the temperatures of the material and of the water are *not the same:*

Specific Gravity $x°/y°$C. .....

where x is the temperature of the material and y is the temperature of the water.

NOTE.—In the interest of standardization and simplification, the first form of statement of specific gravity (*a*) should be employed wherever practicable.

Apparent Specific Gravity (of solids).—The ratio of the weight in air of a given volume of the impermeable portion of a permeable material (that is, the solid matter including its impermeable pores or voids) at a stated temperature to the weight in air of an equal volume of distilled water at a stated temperature. It shall be stated thus:

(*a*) When the temperatures of the material and of the water are the *same:*

Apparent Specific Gravity $x°/x°$C. .....

[1] Under the standardization procedure of the Society, these definitions are under the jurisdiction of the A.S.T.M. Committee E-8 on Nomenclature and Definitions.

[2] Prior to adoption as standard, these definitions were published as tentative from 1925 to 1927, being revised in 1926 and 1927.

[3] Distilled water boiled vigorously in vacuum.

where x is the temperature of the material and the water.

(*b*) When the temperatures of the material and of the water are *not the same:*

Apparent Specific Gravity $x°/y°C.$ .....

where x is the temperature of the material and y is the temperature of the water.

NOTE 1.—In scientific circles, specific gravity and density determinations made in air (that is, uncorrected to vacuum) are frequently distinguished by the adjective "apparent." Thus, the specific gravity defined by the second definition (specific gravity) would be designated as "apparent specific gravity" and that defined by the first definition (absolute specific gravity) as "specific gravity." But in industry, the terminology is more generally in accordance with that given in these definitions.

NOTE 2.—The terms "permeable" and "impermeable" cannot be rigidly defined for general application. The exact meaning in a particular application is the conventional one inferred by the procedure specified for determining the specific gravity of the material in question.

NOTE 3.—In the interest of standardization and simplification, the first form of statement of specific gravity (*a*) should be employed wherever practicable.

Bulk Specific Gravity (of solids).—The ratio of the weight in air of a given volume of a permeable material (including both permeable and impermeable voids normal to the material) at a stated temperature to the weight in air of an equal volume of distilled water at a stated temperature. It shall be stated thus:

(*a*) When the temperatures of the material and of the water are the *same:*

Bulk Specific Gravity $x°/x°C.$ .....

where x is the temperature of the material and the water.

(*b*) When the temperatures of the material and of the water are *not the same:*

Bulk Specific Gravity $x°/y°C.$ .....

where x is the temperature of the material and y is the temperature of the water.

NOTE 1.—See Note 2 under "Apparent Specific Gravity."

NOTE 2.—In the interest of standardization and simplification, the first form of statement of specific gravity (*a*) should be employed wherever practicable.

Standard Definition of

THE TERM SCREEN (SIEVE)[1]

A.S.T.M. Designation: E 13 – 42

ADOPTED, 1942.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation E 13; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Screen (Sieve).—A plate or sheet or a woven cloth, or other device, with regularly spaced apertures of uniform size, mounted in a suitable frame or holder, for use in separating material according to size.

NOTE 1.—The shape and spacing of apertures, size of wires or threads, thickness of plate or sheet, allowable variations and similar properties should be taken care of in specifications.

NOTE 2.—In mechanical analysis testing work, when not otherwise specified, the term "sieve" shall apply to an apparatus in which the apertures are square, and the term "screen" shall apply to an apparatus in which the apertures are circular.

[1] Under the standardization procedure of the Society, this definition is under the jurisdiction of the A.S.T.M. Committee E-8 on Nomenclature and Definitions.

[2] Prior to adoption as standard, this definition was published as tentative from 1925 to 1942, being revised in 1926, 1928, and 1942.

Standard Definitions of

TERMS RELATING TO RHEOLOGICAL PROPERTIES OF MATTER[1]

A.S.T.M. Designation: E 24 – 42

ADOPTED, 1942.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation E 24; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Consistency.—That property of a body by virtue of which it tends to resist deformation.

Plasticity.—That property of a body by virtue of which it tends to retain its deformation after reduction of the deforming stress to its yield stress.

Elasticity.—That property of a body by virtue of which it tends to recover its original size and shape after deformation.

Liquid.—A substance which undergoes continuous deformation when subjected to shearing stress.

Simple Liquid.—A liquid in which the rate of shear is proportional to the shearing stress. The constant ratio of shearing stress to rate of shear of a simple liquid is the *viscosity* of the liquid.

Complex Liquid.—A liquid in which the rate of shear is not proportional to the shearing stress.

Solid.—A substance which undergoes permanent deformation only when subjected to shearing stress in excess of some finite value characteristic of the substance (yield stress).

Plastic Solid.—A substance which does not deform under a shearing stress until the stress attains the yield stress, when the solid deforms permanently.

Elastic Solid.—A substance in which, for all values of the shearing stress below the rupture stress (shear strength), the strain is fully determined by the stress regardless of whether the stress is increasing or decreasing.

[1] Under the standardization procedure of the Society, these definitions are under the jurisdiction of the A.S.T.M. Committee E-1 on Methods of Testing.

[2] Prior to adoption as standard, these definitions were published as tentative from 1934 to 1942, being revised in 1937 and 1940.

A.S.T.M. EMERGENCY STANDARDS

AND

EMERGENCY ALTERNATE PROVISIONS

(*As of November, 1944*)

IN A.S.T.M. STANDARDS AND
TENTATIVE STANDARDS RELATING TO
NONMETALLIC MATERIALS—CONSTRUCTIONAL

(For revisions or change of status in any of these Emergency Standards or Emergency Alternate Provisions, see supplementary publications to be issued in 1945 and 1946.)

CONTENTS

A.S.T.M. EMERGENCY STANDARDS RELATING TO NON-METALLIC MATERIALS—CONSTRUCTIONAL

Emergency Standards and *Emergency Alternate Provisions* are issued by the Society in accordance with a special procedure[1] in the interest of expediting procurement or conservation of materials during the period of National Emergency. They are intended for use where they may be considered by the purchaser of the material as suitable for the specific application or use desired, or as permissible alternates for existing standards. For convenience, the Emergency Alternate Provisions may be cut out and attached to the standards to which they apply.

New and revised Emergency Standards and Emergency Alternate Provisions will appear in the Supplements to the Book of A.S.T.M. Standards. It is important that these Supplements be consulted in case any change of status has occurred with respect to any of the Emergency Standards and Emergency Alternate Provisions.

EMERGENCY STANDARDS

Thermal Insulating Materials

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[1] This special procedure is set forth in the 1944 A.S.T.M. Year Book.

EMERGENCY SPECIFICATIONS
FOR
BLANKET THERMAL INSULATION FOR BUILDING PURPOSES[1]

A.S.T.M. Designation: ES - 14

ISSUED, AUGUST 24, 1942.

Emergency Specifications are issued by the American Society for Testing Materials in accordance with a special procedure[2] in the interest of expediting procurement or conservation of materials during the period of National Emergency. They are intended for use where they may be considered by the purchaser of the material as suitable for the specific application or use desired, or as a permissible alternate for existing specifications.

Scope

1. These specifications cover blanket or batt thermal insulating materials furnished in flexible or semirigid form and suitable for building purposes for application to flat or curved surfaces.

Materials and Manufacture

2. (*a*) The insulation shall consist of clean fibrous material, organic or inorganic, suitably bonded or reinforced or both.

(*b*) The material shall be effectively surfaced or reinforced with one or more sheets of strong durable paper or suitable textile, unless it is of such nature as to make the use of surfacing materials obviously unnecessary.

Physical Properties

3. The blanket insulation shall conform to the following requirements as to physical properties:

Density, max., lb. per cu. ft.	8
Thermal conductivity, max., Btu. in. per sq. ft. per hr. per deg. Fahr., at mean temperature of 75 F.	0.30[a]

[a] Read from the conductivity - mean temperature curve.

Thickness

4. The blanket insulation shall be furnished in thicknesses from ½ to 4 in. in increments of ½ in. No minus variation in thickness shall be permitted.

Special Requirements

5. These specifications are intended to describe adequately the necessary characteristics for a satisfactory insulating material for general building purposes. However, under certain circumstances, other properties, not covered in these specifications, may be important, such as, adequate moisture-vapor resistance, resistance to compression, resilience, odor, effect of liquid water, fire or flame resistance, dustiness, and handling stability. When considered important, limitations on special properties desired shall be as agreed upon by the seller and the purchaser.

[1] Under the standardization procedure of the Society, these Emergency Specifications are under the jurisdiction of the A.S.T.M. Committee C-16 on Thermal Insulating Materials.

[2] This special procedure is set forth in the 1944 A.S.T.M. Year Book.

Sampling

6. (*a*) Samples shall be taken from unbroken packages.

(*b*) Approximately 1 per cent of the number of blankets or batts in a shipment shall be selected at random for purpose of tests, but in no case shall less than two or more than five samples be required.

Methods of Testing

7. The properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(*a*) *Thickness.*—Standard Methods of Test for Thickness and Density of Blanket Type Thermal Insulating Materials (A.S.T.M. Designation: C 167).[3]

(*b*) *Density.*—Standard Methods of Test for Thickness and Density of Blanket Type Thermal Insulating Materials (A.S.T.M. Designation: C 167).[3]

(*c*) *Thermal Conductivity.*—Tentative Method of Test for Thermal Conductivity of Materials by Means of the Guarded Hot Plate (A.S.T.M. Designation: C 177).[3]

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

EMERGENCY SPECIFICATIONS

FOR

BLANKET THERMAL INSULATION FOR INDUSTRIAL PURPOSES[1]

A.S.T.M. Designation: ES – 15

ISSUED, AUGUST 24, 1942.

Emergency Specifications are issued by the American Society for Testing Materials in accordance with a special procedure[2] in the interest of expediting procurement or conservation of materials during the period of National Emergency. They are intended for use where they may be considered by the purchaser of the material as suitable for the specific application or use desired, or as a permissible alternate for existing specifications.

Scope

1. These specifications cover blanket insulating materials furnished in flexible or semirigid form and suitable for industrial purposes for application on flat or curved surfaces where surface temperatures exceed 100 F.

Materials and Manufacture

2. The insulation shall consist of inorganic fibrous materials suitably felted, woven, stitched, bonded, or reinforced.

Physical Properties

3. (*a*) *Thermal Conductivity.*—The thermal conductivity of the material at any given mean temperature shall not exceed the maximum value reported by the manufacturer.

(*b*) *Density.*—The density shall not vary from the density value at which the conductivity was determined by more than plus or minus 10 per cent.

(*c*) *Service Stability.*—The thermal conductivity of the insulating material shall not be increased more than 5 per cent through service of the insulation at any temperature up to the maximum recommended by the manufacturer. Limitations on changes in other properties, resulting from service at any temperature up to the maximum recommended by the manufacturer, shall be as agreed upon by the purchaser and the seller.

Thickness

4. The insulation shall be furnished in thicknesses from 1 to 6 in. in increments of $\frac{1}{2}$ in. Permissible variations in thickness shall not be more than plus or minus $\frac{1}{16}$ in. per inch of thickness.

Sampling

5. (*a*) Samples shall be taken from unbroken packages.

(*b*) Approximately 1 per cent of the number of blankets in a shipment shall be selected at random for purpose of tests, but in no case shall less than two or more than five samples be required.

[1] Under the standardization procedure of the Society, these Emergency Specifications are under the jurisdiction of the A.S.T.M. Committee C-16 on Thermal Insulating Materials.

[2] This special procedure is set forth in the 1944 A.S.T.M. Year Book.

Methods of Testing

6. The properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(*a*) *Thickness.*—Standard Methods of Test for Thickness and Density of Blanket Type Thermal Insulating Materials (A.S.T.M. Designation: C 167).[3]

(*b*) *Density.*—Standard Methods of Test for Thickness and Density of Blanket Type Thermal Insulating Materials (A.S.T.M. Designation: C 167).[3]

(*c*) *Thermal Conductivity.*—Tentative Method of Test for Thermal Conductivity of Materials by Means of the Guarded Hot Plate (A.S.T.M. Designation: C 177).[3]

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

ES – 16

EMERGENCY SPECIFICATIONS

FOR

BLANKET THERMAL INSULATION FOR REFRIGERATION PURPOSES[1]

A.S.T.M. Designation: ES – 16

ISSUED, AUGUST 24, 1942.

Emergency Specifications are issued by the American Society for Testing Materials in accordance with a special procedure[2] in the interest of expediting procurement or conservation of materials during the period of National Emergency. They are intended for use where they may be considered by the purchaser of the material as suitable for the specific application or use desired, or as a permissible alternate for existing specifications.

Scope

1. These specifications cover blanket thermal insulating materials furnished in flexible or semirigid form and suitable for refrigeration purposes for application on flat or curved surfaces.

Materials and Manufacture

2. (*a*) The insulation shall consist of clean fibrous material, organic or inorganic, suitably bonded or reinforced or both.

(*b*) The material shall be effectively confined between sheets of strong durable paper or suitable textile, unless it is of such a nature as obviously to make the use of confining members unnecessary. When specified, the paper shall be waterproofed.

Physical Properties

3. The blanket insulation shall conform to the following requirements as to physical properties:

Density, max., lb. per cu. ft.	7
Thermal conductivity, max., Btu. in. per sq. ft. per hr. per deg. Fahr., at mean temperature of 75 F.	0.32[a]

[a] Read from the conductivity - mean temperature curve.

Thickness

4. The blanket insulation shall be furnished in thicknesses from ½ to 4 in. in increments of ½ in. No minus variation in thickness shall be permitted.

Special Requirements

5. These specifications are intended to describe adequately the necessary characteristics for a satisfactory material for general refrigeration purposes. However, under certain circumstances, other properties, not covered in these specifications, may be important, such as, specific heat, odor, resistance to compression, resilience, dustiness, content of nonfibrous particles, and resistance to disintegration by water. When considered important, limitations on special properties desired shall be as agreed upon by the seller and the purchaser.

[1] Under the standardization procedure of the Society, these Emergency Specifications are under the jurisdiction of the A.S.T.M. Committee C-16 on Thermal Insulating Materials.

[2] This special procedure is set forth in the 1944 A.S.T.M. Year Book.

Sampling

6. (*a*) Samples shall be taken from unbroken packages.

(*b*) Approximately 1 per cent of the number of blankets in a shipment shall be selected at random for purpose of tests, but in no case shall less than two or more than five samples be required.

Methods of Testing

7. The properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(*a*) *Thickness.*—Standard Methods of Test for Thickness and Density of Blanket Type Thermal Insulating Materials (A.S.T.M. Designation: C 167).[3]

(*b*) *Density.*—Standard Methods of Test for Thickness and Density of Blanket Type Thermal Insulating Materials (A.S.T.M. Designation: C 167).[3]

(*c*) *Thermal Conductivity.*—Tentative Method of Test for Thermal Conductivity of Materials by Means of the Guarded Hot Plate (A.S.T.M. Designation: C 177).[3]

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

EMERGENCY SPECIFICATIONS
FOR
PREFORMED PIPE COVERING THERMAL INSULATION[1]

A.S.T.M. Designation: ES – 17

ISSUED, AUGUST 24, 1942.

Emergency Specifications are issued by the American Society for Testing Materials in accordance with a special procedure[2] in the interest of expediting procurement or conservation of materials during the period of National Emergency. They are intended for use where they may be considered by the purchaser of the material as suitable for the specific application or use desired, or as a permissible alternate for existing specifications.

Scope

1. These specifications cover preformed (molded or laminated) thermal insulation in the form of semicylindrical pipe covering sections intended for use as an insulation on surfaces at specified temperatures.

NOTE.—These specifications do not include materials ordinarily used in the refrigerating temperature range.

Basis of Purchase

2. Orders for material under these specifications shall include the following information:

(*1*) Temperature range for which pipe covering is to be used,

(*2*) Pipe size,

(*3*) Thickness of insulation,

(*4*) Jacketing or banding desired, or both.

Materials and Manufacture

3. (*a*) *Diatomaceous Silica* pipe insulating material shall be composed predominantly of diatomaceous silica and long-fiber asbestos and may contain minor proportions of basic magnesium carbonate, calcium carbonate, clay, or other suitable heat-resistant mineral fillers and binders.

(*b*) *85 per cent Magnesia* pipe insulating material shall be composed of a minimum of 85 per cent, by weight, of basic hydrated magnesium carbonate and a minimum of 10 per cent of long-fiber asbestos.

(*c*) *Laminated, and Laminated and Indented* pipe insulating material shall be composed of layers of organic or inorganic sheets that have been formed into semicylindrical sectional pipe covering form by winding on a mandrel to the desired number of laminations or layers, held in position by suitable binders that, when combined in this insulation, are satisfactory up to the temperatures for which the pipe covering is recommended, as shown in Table I. Certain insulating particles or indentations of the sheet may be included in order to separate the laminations.

(*d*) *Corrugated* pipe insulating material shall be composed of layers of in-

[1] Under the standardization procedure of the Society, these Emergency Specifications are under the jurisdiction of the A.S.T.M. Committee C-16 on Thermal Insulating Materials.

[2] This special procedure is set forth in the 1944 A.S.T.M. Year Book.

organic corrugated sheets that have been formed into semicylindrical pipe covering sections by winding on a mandrel to the desired number of layers, held in position by suitable binders that, when combined in this insulation, are satisfactory up to the temperature for which the pipe covering is recommended, as shown in Table I.

(*e*) *Fibrous* pipe insulating materials shall be composed predominantly of fibrous asbestos or mineral wool and may contain minor proportions of suitable organic or inorganic fillers and binders that, when combined in this insulation, are satisfactory up to the temperature for which the pipe covering is recommended, as shown in Table I.

(*f*) *Vermiculite* pipe insulating material shall be composed predominantly of expanded or exfoliated mica and long-fiber asbestos and may contain minor proportions of suitable organic or inorganic fillers and binders that, when combined with this insulation, are satisfactory up to the temperatures for which the pipe covering is recommended, as shown in Table I.

(*g*) *Cork* pipe insulating material shall be composed predominantly of granulated cork and its natural resins.

Physical Properties

4. The preformed pipe covering shall conform to the requirements as to physical properties prescribed in Table I.

Sampling

5. For purpose of tests, pipe sections that are representative of the commercial product shall be selected by the representative of the purchaser. The point of selection shall be designated at the time of placing the order. The pipe sections shall be systematically obtained, selecting a number of pipe sections equal to one fifth the cube root of the total nunber of pipe sections in the shipment, but not less than four pipe sections, and in any event a sufficient number to cover all the required tests.

TABLE I.—PHYSICAL REQUIREMENTS.[a]

Material	Temperature Range, deg. Fahr.	Density, max., lb. per cu. ft.	Linear Heat Shrinkage	Thermal Conductivity, Btu. in per sq. ft. per hr. per deg. Fahr.[b]
Diatomaceous silica (molded)	1200 to 1900	30	5 per cent after 24 hr. at 1900 F.	1.10 at 1000 F.
	600 to 1200	30	5 per cent after 24 hr. at 1900 F.	0.80 at 500 F.
Magnesia (85 per cent)	212 to 600	17	.....	0.60 at 300 F.
Laminated and laminated and indented:				
Inorganic	212 to 600	43	.....	0.60 at 300 F.
Organic	33 to 212	40	.....	0.50 at 100 F.
Corrugated				
Inorganic	212 to 600	25	.....	0.70 at 200 F.
Fibrous (molded)	600 to 1200	30	5 per cent after 24 hr. at 1200 F.	0.80 at 500 F.
	212 to 600	22	2 per cent after 24 hr. at 600 F.	0.60 at 300 F.
Vermiculite	600 to 1200	20	5 per cent after 24 hr. at 1200 F.	0.90 at 500 F.
	212 to 600	20	3 per cent after 24 hr. at 600 F.	0.80 at 300 F.
	33 to 212	20	none after 24 hr. at 121 F.	0.55 at 100 F.
Cork	33 to 212	12	.....	0.30 at 90 F.

[a] The values given in this table are approximate only and do not necessarily represent any one manufacturer's product.
[b] Read from the conductivity - mean temperature curve.

Methods of Testing

6. The properties enumerated in these specifications shall be determined in accordance with the following methods of test:

(*a*) *Density.*—Four full-size pipe sections shall each be weighed and measured after drying at 225 ± 25 F. in a vented oven for not less than 16 hr. If the material is one that may be chemically or physically affected by heating to 225 ± 25 F., the samples shall be dried in a desiccator at 120 to 140 F. Inside diameter, outside diameter, and length shall be measured to the nearest $\frac{1}{32}$ in. with a steel rule, at least four measurements of each dimension being taken. Thickness shall be taken as the average outside diameter minus the average inside diameter, divided by two. The volume and density of each specimen shall be calculated from the dimensions and weights above determined. The average density value for the four specimens shall be reported.

(*b*) *Thermal Conductivity.*—Tentative Method of Test for Thermal Conductivity of Materials by Means of the Guarded Hot Plate (A.S.T.M. Designation: C 177),[2] except that the test specimen shall be a flat block of material of the same quality and density as the pipe covering section in question. When flat block material of the same quality and density is not available, the method of testing shall be agreed upon by the manufacturer and the purchaser. Thermal conductivity values shall be stated by the manufacturer for each material.

[2] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

EMERGENCY SPECIFICATIONS
FOR
PREFORMED BLOCK THERMAL INSULATION[1]

A.S.T.M. Designation: ES – 18

ISSUED, AUGUST 24, 1942.

Emergency Specifications are issued by the American Society for Testing Materials in accordance with a special procedure[2] in the interest of expediting procurement or conservation of materials during the period of National Emergency. They are intended for use where they may be considered by the purchaser of the material as suitable for the specific application or use desired, or as a permissible alternate for existing specifications.

Scope

1. (*a*) These specifications cover preformed thermal insulating block as defined in the Standard Definitions of Terms Relating to Thermal Insulating Materials (A.S.T.M. Designation: C 168) of the American Society for Testing Materials,[3] namely, "A rigid or semirigid thermal insulating material, either flat or segmental, for application as received, and excluding brick of the 9-in. series." Three classes of block are covered, as follows:

Class A.—Compressive strength less than 5 psi.

Class B.—Compressive strength of 5 to 20 psi.

Class C.—Compressive strength greater than 20 psi.

(*b*) When block are required having physical strength or heat resistance different from those prescribed by these specifications, the purchaser should specify the desired minimum values.

Sampling

2. For purpose of tests, block that are representative of the commercial product shall be selected by the representative of the purchaser, the place or places of selection to be designated when the purchase order is placed. The block shall be systematically obtained, selecting a number of block equal to the fourth root of the total number of block in the shipment, but in any event not less than seven block. When thermal conductivity tests are not required by the purchaser, not less than four block shall be selected.

Density

3. (*a*) All of the samples selected shall be weighed before and after drying at 225 ± 25 F. in a vented oven for not less than 16 hr. and shall be tested promptly after removal from the oven. If the material is one that may be chemically or physically affected by heating at 200 to 250 F., the samples shall be dried in a desiccator at 120 to 140 F.

(*b*) After drying in the oven, the samples shall be measured for length, width, and thickness to within an ac-

[1] Under the standardization procedure of the Society, these Emergency Specifications are under the jurisdiction of the A.S.T.M. Committee C-16 on Thermal Insulating Materials.

[2] This special procedure is set forth in the 1944 A.S.T.M. Year Book.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

curacy of plus or minus 1 per cent. The volume of each sample and its density shall be calculated from the dimensions and weights after drying. The density value reported shall be the average of all values obtained.

(*c*) From all the test samples selected in accordance with Section 2, the three samples selected for thermal conductivity tests, when specifically requested by the purchaser, shall be one each of high, average, and low density.

(*d*) The four samples selected for flexural, compressive, and heat-resistance tests shall be one each of high and low density and two of average density.

Flexural Strength

4. The flexural strength of the insulating block, when determined in accordance with the Tentative Methods of Test for Compressive Strength and Flexural Strength of Preformed Block Type Thermal Insulating Materials (A.S.T.M. Designation: C 165) of the American Society for Testing Materials,[3] shall be not less than that calculated as follows:

$$R' = \frac{3.6\rho l^2}{144\, d}$$

where:

R' = Flexural strength in pounds per square inch,
ρ = Average density in pounds per cubic foot,
l = Length of test specimen in inches, and
d = Thickness of test specimen in inches.

Compressive Strength

5. The compressive strength of the insulating block, when determined in accordance with Tentative Methods C 165,[3] shall conform to the following requirements:

	Compressive Strength, psi.
Class A	under 5
Class B	5 to 20
Class C	over 20

Heat Resistance

6. The requirements for heat resistance shall apply only to block insulation used at temperatures higher than that of the normal atmosphere. The upper useful temperature for which a material is recommended to the purchaser shall conform to the following requirements:

The change in length and width of the test specimen after subjecting it to soaking heat at the recommended useful temperature for 24 hr. shall not be greater than 3 per cent. The test specimens shall be not less than 1½ by 3 by 6 in. The average value obtained from four specimens shall be reported. Compressive strength of test specimens after subjecting them to a soaking heat for 24 hr. at the maximum recommended useful temperature shall be not less than 75 per cent of the strength as determined at room temperature for class A and class B block and not less than 15 psi. for class C block. The specimens used for compressive strength tests at room temperature shall be cut from the same samples as are those subjected to soaking heat. Four specimens from each sample shall be tested at room temperature and four at the recommended useful temperature and the average value obtained from each group of four specimens shall be reported.

Thermal Conductivity

7. Thermal conductivity tests shall be made only when specifically requested by the purchaser. When specified, they shall be made at the mean temperatures agreed upon by the seller and the purchaser in accordance with the Tentative Method of Test for Thermal Conductiv-

ity of Materials by Means of the Guarded Hot Plate (A.S.T.M. Designation: C 177) of the American Society for Testing Materials.[3] The highest thermal conductivity of the samples shall deviate from the average curve supplied by the manufacturer or seller not more than the maximum deviation reported with the average curve.

Permissible Variations in Dimensions

8. (*a*) The insulating block shall be true to form and dimensions, the corners square, and the sides and ends parallel. Permissible variations in dimensions shall be as follows:

Dimension	Permissible Variations, in.
Thickness	± 1/8
Width	± 1/8
Length	± 1/8

(*b*) For insulating block that are to be used on surfaces where the temperature is below the surrounding air temperature, permissible variations in dimensions shall be as follows:

Dimension	Permissible Variations, in.
Thickness	± 1/16
Width	± 1/16
Length	± 1/8

Damage Tolerance

9. The number of broken or damaged blocks shall not exceed 5 per cent of any one shipment.

EMERGENCY SPECIFICATIONS

FOR

STRUCTURAL INSULATING BOARD (THERMAL INSULATION)[1]

A.S.T.M. Designation: ES – 19

ISSUED, AUGUST 24, 1942.

Emergency Specifications are issued by the American Society for Testing Materials in accordance with a special procedure[2] in the interest of expediting procurement or conservation of materials during the period of National Emergency. They are intended for use where they may be considered by the purchaser of the material as suitable for the specific application or use desired, or as a permissible alternate for existing specifications.

Scope

1. These specifications cover thermal insulating material made principally of vegetable fiber, in the form of insulating board suitable for structural purposes. Five classes of insulating board are covered, classified according to use as follows:

Class A.—Boards for uses other than for classes B, C, D, and E.

Class B.—Lath (for plaster base).

Class C.—Roof board.

Class D.—Interior boards (factory finished).

Class E.—Sheathing.

Definition

2. A board insulation is a preformed, rigid, fibrous material used principally in building construction and having the following properties:

(*1*) A thermal conductivity k of not more than 0.50 Btu. in. per sq. ft. per hr. per deg. Fahr.,

(*2*) A thermal resistance of not less than 1.0 deg. Fahr. per Btu. per hr., and

(*3*) A minimum load at rupture of 7 lb.

Physical Properties

3. The board insulation shall conform to the requirements as to physical properties prescribed in Table I.

Dimensions

4. The board insulation shall conform to the requirements for dimensions prescribed in Table II.

Permissible Variations in Dimensions

5. The following variations in dimensions of the board insulation are permissible:

Dimension	Permissible Variations, in. Plus	Minus
Length	none	$\frac{1}{4}$
Width	none	$\frac{1}{4}$
Thickness	$\frac{1}{16}$	$\frac{1}{16}$

Edge Finish

6. Edge finish shall be as follows:

Class A.—Edges shall be square.

Class B.—Edges shall be either shiplapped, beveled and shiplapped, tongue and grooved, beveled and tongue and

[1] Under the standardization procedure of the Society, these Emergency Specifications are under the jurisdiction of the A.S.T.M. Committee C-16 on Thermal Insulating Materials.

[2] This special procedure is set forth in the 1944 A.S.T.M. Year Book.

grooved, V-jointed, or shiplapped and grooved.

Class C.—Edges shall be square unless shiplapped or offset are specified. When offset boards 1½ in. and over in thickness are specified, the offset shall be one half the thickness of the board.

Class D.—Edges shall be either square, shiplapped, V-jointed, or shiplapped and grooved.

Class E.—Edges shall be either square, shiplapped, beveled and shiplapped, tongue and grooved, V-jointed, or shiplapped and grooved.

Surface Finish

7. For plaster base, roof boards, and similar applications, surfaces shall be natural and shall be free of cracks, lumps, excessive departure from planeness, or other defects. For interior finish, partitions, and similar applications where the board is exposed, surfaces shall be finished and may be textured or smooth and reasonably free of coarse and hairy fibers.

Sampling

8. Samples shall be taken at random so as to give a fair representation of the entire shipment. Pieces from five boards of any carload, or 0.5 per cent of the number of pieces in less than carload shipments but not less than three boards of any shipment, or not less than sufficient area to meet test requirements, shall constitute the sample. From each board of the sample a specimen 24 by 30 in. shall be cut, the longer dimension of the specimen being crosswise of the longer dimension of the board as it is usually supplied. Where the short dimension of the boards making up the sample is less than 30 in., the specimen shall be cut to 30 in. in length by the short dimension; and where the over-all size of the board making up the sample is 24 by 48 in., or less, sufficient full-size pieces to satisfy test requirements shall be the specimen. Where a shipment consists of more than one car or carrier load, a sample shall be selected from each car or carrier.

TABLE I.—PHYSICAL REQUIREMENTS.

	Class A	Class B	Class C	Class D	Class E
Thermal conductivity, max., Btu. in. per sq. ft. per hr. per deg. Fahr.	0.50	0.38	0.38	0.38	0.40
Load at rupture, min., lb.[a]	12	12	7	10	14
Deflection under specified minimum load at rupture, max., in.	0.85	0.85	1.25	1.00	0.75
Tensile strength, min., psi.[b]	175	175	100	130	130
Water absorption, max., per cent by volume	7	7	10	10	7
Linear expansion, max., per cent	0.5	0.5	0.5	0.5	0.5

[a] The requirement is based on specimens ½ in. in thickness. For specimens of other thicknesses, the requirement shall be increased in direct proportion, that is, for a thickness of 1 in. it shall be doubled, etc.
[b] Tensile strength requirements shall be applicable only on thicknesses up to and including 1 in.

TABLE II.—REQUIREMENTS FOR DIMENSIONS.

	Widths	Lengths	Thicknesses
Class A	4 ft.	6, 7, 8, 9, 10, and 12 ft.	½, ¾, ⅞, and multiples of ½ in.
Class B	16, 18, and 24 in.	48 in.	multiples of ½ in.
Class C	22 and 23 in.	47 in.	multiples of ½ in.
	24 in.	48 in.	
Class D:			
Panels	8 in.	8 in.	½, ¾, and 1 in.
	12 in.	12 and 24 in.	
	16 in.	16 and 32 in.	
Planks	8, 10, 12, and 16 in.	6, 8, 10, and 12 ft.	½, ¾, and 1 in.
Class E	4 ft.	8, 8½, 9, 9½, 10, or 12 ft.	½ and ⅝ in.
	2 ft.	8 ft.	⅝ in.

Methods of Testing

9. The properties enumerated in these specifications shall be determined in accordance with the following methods of test:

(*a*) *Thermal Conductivity.*—Tentative Method of Test for Thermal Conductivity of Materials by Means of the Guarded Hot Plate (A.S.T.M. Designation: C 177) of the American Society for Testing Materials.[3]

(*b*) *Load at Rupture.*[4]—A specimen 3 in. in width and 18 in. in length shall be taken for test. The transverse breaking load shall be determined by placing the specimen on horizontal parallel supports 12 in. apart and applying the load at midspan on a bearing parallel to the end supports, so that the head of the testing machine through which the load is applied moves at the rate of 2 in. per min. until the specimen breaks. The bearing and supports shall be rounded to a radius of approximately $\frac{3}{8}$ in. to prevent injury to the specimen. Three specimens from the long direction of the board and three at right angles thereto shall be tested. The transverse breaking load in each direction is the average of three specimens taken from that direction.

NOTE.—See *Miscellaneous Publication No. 132*, National Bureau of Standards, Issued December 1, 1931.

(*c*) *Deflection at Rupture.*[5]—Maximum deflection shall be determined on specimens tested in accordance with Paragraph (*b*), at the time the specimen is under the specified minimum transverse load, as given in Table I.

(*d*) *Tensile Strength.*[6]—From each sample, specimens 2 in. in width and not less than 10 in. in length shall be cleanly cut parallel to the longest direction of the board and at right angles thereto. The distance between clamps shall be not less than 6 in. Results on specimens under test that break within $\frac{1}{2}$ in. of the jaws shall be disregarded. The machine speed shall be set for the openings between the clamps at the rate of 2 in. per min. The specimens before being placed in the testing machine shall be measured for width and thickness to the nearest 0.01 in. The tensile strength shall be taken as the average of three specimens from each direction of the board.

(*e*) *Water Absorption.*[7]—A 12-in. square specimen shall be dried at 160 F. for 24 hr. and cooled to room temperature in a dry atmosphere. The thickness of the sample shall be measured and the volume calculated therefrom. The sample shall then be carefully weighed and submerged horizontally under 1 in. of distilled water maintained at a temperature of 70 ± 5 F. After submersion for 2 hr., the sample shall be stood on end to drain for 10 min., at the end of which time the excess surface water shall be removed by hand with a blotting paper or paper towel and the sample immediately weighed, the volume of absorbed water calculated, and the water absorption expressed in percentage by volume based on the initial volume.

(*f*) *Linear Expansion.*[8]—The maximum linear expansion shall be determined from a specimen 3 by 12 in. that has been cut parallel with the long

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

[4] This test for load at rupture is substantially identical with Paragraph F-2c of the Federal Specification for Fiberboard; Insulating (LLL-F-321b), except that deflection shall be at the rate of 2.0 in. per min.

[5] This test for deflection at rupture is substantially identical with Paragraph F-2d of the Federal Specification for Fiberboard; Insulating (LLL-F-321b), except that only deflection at the time the specimen is under the specified minimum transverse load, as given in Table I, is determined and deflection shall be at the rate of 2.0 in. per min.

[6] This test for tensile strength is substantially identical with Paragraph F-2e of the Federal Specification for Fiberboard; Insulating (LLL-F-321b).

[7] This test for water absorption is substantially identical with Paragraph F-2f of the Federal Specification for Fiberboard; Insulating (LLL-F-321b), except that all edges of specimens shall be freshly cut.

[8] This test for linear expansion is substantially identical with Paragraph F-2h of the Federal Specification for Fiberboard; Insulating (LLL-F-321b).

direction of the board and from a like specimen cut at right angles thereto. At each of two points approximately 10 in. apart on the center line of each specimen a small area shall be coated by rubbing with a glass marking pencil (or a wax crayon). A fine cross mark shall be made with a razor blade on the center line in each of these two areas as reference points for the length measurements. The specimens then shall be conditioned for 24 hr. in an atmosphere at 50 per cent relative humidity and measurements made of the distance between the two reference points. A scale graduated in hundredths of an inch shall be used and readings made with a magnifying lens to the nearest 0.005 in. The specimens next shall be conditioned for 24 hr. at 97 per cent relative humidity, after which the distance between the two reference points shall be again measured. The measurements shall be made in the conditioned air specified in each case, or as quickly as possible after each specimen is removed therefrom. The linear expansion shall be reported as the increase in the length between reference marks as a percentage of the length at 50 per cent relative humidity. (In the absence of other facilities, a convenient means of conditioning the specimens at the specified humidity conditions is to expose them in an enclosed space immediately above saturated solutions of suitable salts: sodium dichromate ($Na_2Cr_2O_7 \cdot 2H_2O$) for 50 per cent relative humidity, and potassium sulfate (K_2SO_4) for 97 per cent relative humidity.

Inspection

10. Inspection may be made either at the point of shipment or at the point of delivery. The inspector representing the purchaser shall have free access to the carrier being loaded for shipment to the purchaser. He shall be afforded all reasonable facilities for inspection and sampling, which shall be so conducted as not to interfere unnecessarily with the loading of the carriers.

Rejection

11. Any rejection shall be based upon the specific failure to conform to these specifications and shall be reported to the seller within ten working days from the receipt of the shipment by the purchaser.

EMERGENCY METHOD OF TEST

FOR

CHANGES IN PROTECTIVE PROPERTIES OF ORGANIC COATINGS ON STEEL SURFACES WHEN SUBJECTED TO IMMERSION[1]

A.S.T.M. Designation: ES – 35

ISSUED, JANUARY 31, 1944.

Emergency Methods are issued by the American Society for Testing Materials in accordance with a special procedure[2] in the interest of expediting procurement or conservation of materials during the period of National Emergency. They are intended for use where they may be considered by the purchaser of the materials as suitable for the specific application or use desired, or as a permissible alternate for existing methods.

Scope

1. This method of test is intended for use in determining the resistance to failure of organic coatings when immersed in specified test liquids. It may be used on coated steel panels and on coated steel manufactured articles.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Container.*—A suitable container made of corrosion-proof material, such as glass, earthenware, porcelain-coated metal, or similar material. It shall be sufficiently large to accommodate the test panels or specimens at a spacing of not less than $\frac{1}{2}$ in. between adjacent panels or specimens. Provision shall be made for maintaining the temperature within the range specified.

(*b*) *Test Liquid.*—The test liquid used in the container, whether aqueous or nonaqueous, shall be as agreed upon by the purchaser and the seller. Tap water may be used but, because of its variable composition, the use of distilled water is preferred.

Test Specimens

3. The test specimens may be either coated steel panels or samples of the coated product. Test panels shall be prepared from new cold-rolled steel (No. 20 to No. 22 gage) that has been oiled at the mill and is free from rust. The steel shall conform to the Standard Specifications for Cold-Rolled Strip Steel (A.S.T.M. Designation: A 109),[3] and shall have a No. 5 dead soft temper and a No. 1 or dull finish.[4] The panels shall be not less than $2\frac{3}{4}$ by $5\frac{7}{8}$ in. in area and all edges and corners shall be smooth and uniformly rounded.

Preparation of Specimens

4. (*a*) *Coated Products.*—When coated products are used for test specimens the

[1] Under the standardization procedure of the Society, this Emergency Method is under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] This special procedure is set forth in the 1944 A.S.T.M. Year Book.

[3] 1944 Book of A.S.T.M. Standards, Part I.

[4] This finish is also known as No. 60 Pangborn finish; see "Steel Products Manual," Section 13 on Cold-Rolled Carbon Steel Strip, Am. Iron and Steel Inst., July, 1942.

preparation shall be that used in production.

(*b*) *New Test Panels.*—Test panels not previously used shall be prepared for painting by one of the two following procedures *A* or *B* (Note 1):

(*1*) *Procedure A, Cleaning.*—The test panels shall be cleaned without sanding or abrasion of the surface by the use of suitable grease-removing solvents (toluol, light naphtha, or trichlorethylene type vapors) so that the surface shows no water break when immersed in distilled water. The panels shall then be immersed in alcohol (ethyl, isopropyl, or suitable denatured alcohol), removed, and allowed to dry at a temperature between 125 and 200 F. The panels shall then be cooled to room temperature in a desiccator and coated, or they may be stored in a desiccator until ready for finishing.

(*2*) *Procedure B, Pretreatment.*—The test panels shall be cleaned without sanding or abrasion of the surface by the use of suitable grease-removing solvents (toluol, light naphtha, or trichlorethylene type vapors) so that the surface shows no water break when immersed in distilled water. The panels shall then be subjected to a phosphate solution until a uniform phosphate coating is obtained. The conditions of the phosphate treatment shall be agreed upon by the purchaser and the seller (Note 2). The panels shall then be coated, or they may be stored in a desiccator until ready for finishing.

NOTE 1.—When used or rusty panels free from paint (rust pitted or scarred panels shall be discarded) are to be reconditioned, all rust or corrosion spots shall be removed by sanding with No. 240 sandpaper or equal grade emery cloth. The panels shall then be lightly buffed (using steel wool and lacquer thinner), rinsed with lacquer thinner or light naphtha (using a soft bristle brush) and wiped dry with a clean rag. The panels shall then be degreased in trichlorethylene or tetrachlorethylene (perchlorethylene) vapor for 5 min. When a vapor cleaning machine is not available, a cold dip in chlorethylene solvent or carbon tetrachloride may be used. In this case, the solvent shall be changed frequently, and the panels shall be rinsed with fresh solvent. If not coated immediately, provision shall be made for storage of the panels under conditions that will prevent corrosion.

NOTE 2.—When phosphate-treated panels are required, the panels (previously cleaned as described in Note 1) shall be immersed for 1 min. at room temperature in phosphate cleaner of the following composition:

Syrupy phosphoric acid (85 per cent).....	3 ml.
Penetrant[5].....	1 ml.
Water.....to bring the volume to	1000 ml.

The panels shall then be drained for a few seconds in a vertical position and dried at 250 F. for 5 min. after which they may be coated or stored under conditions that will not cause corrosion.

Coating of Specimens

5. The organic coating shall be applied at a specified film thickness or film thickness range by the specified method, such as brush, spray, dip coat, flow-out, or other method. The film thickness shall be measured by the usual methods. When panels are used, the backs and edges of the panels shall be completely protected, each with its individual organic test coating (Note). The drying time and conditions, or baking schedule prior to immersion, shall be as prescribed in the specifications.

NOTE.—The relative merits of immersion tests with fully coated panels *versus* panels with the test coating on one side only and the other side uncoated is a subject still under discussion. There is evidence that breaks which develop in the coating or bare areas on one side of a panel influence the degree of blistering on the other side. While the reproducibility of immersion tests made with panels coated on one side only appears satisfactory, the relative significance of the results compared with articles of manufacture that are completely coated is still subject to question and for this reason coating with the test paint on all sides and edges is preferred. Unless edge failure is of specific interest, the

[5] Tergitol penetrant No. 4 or equivalent is satisfactory for this purpose.

edges should be given additional protection in the form of extra coats of paint, or, if a cold water immersion test is to be made, paraffin may be used.

Procedure

6. (*a*) *Immersion.*—After being dried for the specified length of time, the specimens shall be partially immersed in the test liquid, which shall be maintained at a temperature of 25 ± 2 C. or other mutually agreed upon temperature. Panels having uniform film thickness shall be immersed one-half to two-thirds of their length but panels having graduated film thickness, such as flow-outs, shall be immersed one-half of their width. The panels shall be so arranged that no electrical contact will be obtained between them. A space of not less than $\frac{1}{2}$ in. shall be left between adjacent panels. The test liquid shall be changed at least every 72 hr. to avoid contamination, or more often if visible rust appears in the liquid. Reference panels coated with a control paint shall be used in order to correlate successive tests with each other.

(*b*) *Examination of Panels.*—When the specimens are ready for examination, they shall be dried by blowing with air or by other specified methods that will remove surface liquid only. Examination shall be made for blistering and wrinkling within 5 min. after removal from the liquid, and ratings for other defects shall follow immediately (Note 1). When ratings for the degree of recovery from immersion effects are specified, the specimens shall be allowed to stand at room temperature, or other prescribed condition, and re-examined after 2 hr. and after 24 hr., or as specified. If re-immersion is necessary, the specimens shall be allowed to remain out of the liquid for the same period of time which, unless otherwise specified, shall not exceed $\frac{1}{2}$ hr. Comparison shall be made between the immersed portion and that which remained completely out of the test liquid. Gradings shall be made on both the fronts and backs of the panels and the results averaged. Unless otherwise specified, failure within $\frac{1}{2}$ in. of the edges shall be disregarded. The test coating shall be examined for the effects of failures specifically agreed upon by the purchaser and the seller (Note 2). Rating for blistering shall be made in accordance with the Tentative Method for Evaluating the Degree of Resistance to Blistering of Organic Coatings on Metal When Subjected to Immersion or Other Tests Involving Exposure to Moisture or Liquids (A.S.T.M. Designation: D 714).[6] Ratings for other failures shall be made by methods mutually agreed upon, or by available A.S.T.M. methods.

NOTE 1.—When a large series of specimens is to be examined, groups that can be handled within the prescribed period of time should be removed in relays.

NOTE 2.—Blistering, wrinkling or roughening, disintegration, loss of adhesion, changes in color, or other specific failures, are usually included in such examination.

(*c*) *Length of Test.*—The test shall be continued for a specified period of time or until a specified degree of failure has occurred.

[6] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

EA-D 147

EMERGENCY ALTERNATE PROVISION

IN

STANDARD METHODS OF TESTING BITUMINOUS MASTICS, GROUTS, AND LIKE MIXTURES

(D 147-41)[1]

ISSUED, AUGUST 30, 1943.

This Emergency Alternate Provision is issued by the American Society for Testing Materials in accordance with a special procedure[2] in the interest of expediting procurement or conservation of materials during the period of National Emergency. It is intended for use where it may be considered by the purchaser of the material as a permissible alternate for the specific application or use desired.

The following Emergency Alternate Provision, when specified, may be used as an alternate in A.S.T.M. Standard Methods of Testing Bituminous Mastics, Grouts, and Like Mixtures (D 147 - 41) and affects only the requirements referred to:

Section 3 (a).—To provide for the use of an alternative extraction apparatus made of tinned copper and having a glass condenser as shown in the accompanying Fig. 1a, change this section to read as follows by the addition of the italicized words and figures:

(OVER)

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[1] This Emergency Alternate Provision applies to the standard appearing on p. 691 and for convenience may be cut out and attached to the standard.

[2] This special procedure is set forth in the 1944 A.S.T.M. Year Book.

3. (*a*) For Analysis of 10 to 30-g. Samples.—In cases where a 10 to 30-*g.* sample is sufficient, the analysis shall be carried out by means of the glass extractor, shown in Fig. 1 *or the accompanying Fig. 1a.*

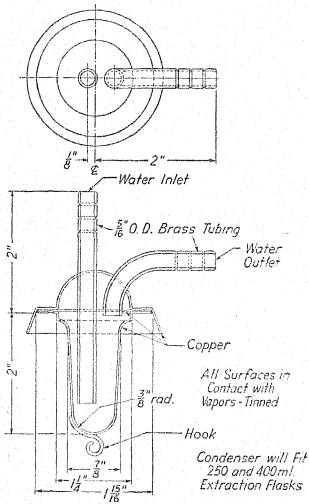


FIG. 1a.—Extraction Apparatus for Analysis of 10 to 30-g. Samples.

EA – D 224b

EMERGENCY ALTERNATE PROVISION

IN

TENTATIVE SPECIFICATIONS FOR ASPHALT ROOFING SURFACED WITH POWDERED TALC OR MICA

(D 224 – 44 T)[1]

ISSUED, AUGUST 28, 1944.

(SUPERSEDING ISSUES OF MAY 11, 1942 AND AUGUST 30, 1943.)

This Emergency Alternate Provision is issued by the American Society for Testing Materials in accordance with a special procedure[2] in the interest of expediting procurement or conservation of materials during the period of National Emergency. It is intended for use where it may be considered by the purchaser of the material as a permissible alternate for the specific application or use desired.

The following Emergency Alternate Provision, when specified, may be used as an alternate in A.S.T.M. Tentative Specifications for Asphalt Roofing Surfaced with Powdered Talc or Mica (D 224 – 44 T) and affects only the requirements referred to:

Section 12 (*a*).—Omit the requirements for protective coating on nails prescribed in item (*2*) of this section.

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[1] This Emergency Alternate Provision applies to the standard appearing on p. 1427 and for convenience may be cut out and attached to the standard.

[2] This special procedure is set forth in the 1944 A.S.T.M. Year Book.

EMERGENCY ALTERNATE PROVISIONS

IN

TENTATIVE SPECIFICATIONS FOR ASPHALT ROOFING SURFACED WITH COARSE MINERAL GRANULES

(D 249 – 44 T)[1]

ISSUED, AUGUST 28, 1944.

(SUPERSEDING ISSUES OF MAY 11, 1942 AND AUGUST 30, 1943.)

These Emergency Alternate Provisions are issued by the American Society for Testing Materials in accordance with a special procedure[2] in the interest of expediting procurement or conservation of materials during the period of National Emergency. They are intended for use where they may be considered by the purchaser of the material as a permissible alternate for the specific application or use desired.

The following Emergency Alternate Provisions, when specified, may be used as an alternate in A.S.T.M. Tentative Specifications for Asphalt Roofing Surfaced with Coarse Mineral Granules (D 249 – 44 T) and affect only the requirements referred to:

Section 6 (*b*).—Change to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

(*b*) The coating and granules shall cover the entire weather side of the roofing, except that this side may be provided with a bare lapping edge, approximately 2 or *approximately* [3] *4* in. in width along one edge, where either the granules alone or both the granules and coating may be omitted. When the granules alone are omitted, [a thin fibrous membrane may be used to prevent the coated lapping edge from sticking in the roll] *any suitable treatment may be applied to the coated surface which shall prevent sticking of the coated lapping edge in the roll and which shall also permit the roofing to pass the strength-of-lap test.*

Section 6 (*c*).—Change to read as follows by the omission of the word in brackets:

(*c*) The reverse side of the sheet shall have the asphalt coating and [powdered] surfacing applied uniformly to the edges.

Table I.—Change the requirements for area to read as follows by the addition of the italicized figures and the omission of those in brackets:

Area.....The average area of the rolls examined shall contain sufficient roofing to cover 100 sq. ft. of roof surface (108 sq. ft. with 2 in. or no bare lapping edge, or [111] *114* sq. ft. with [3] *4* in. lapping edge).

Change the minimum requirements for net average weight per roll

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[1] These Emergency Alternate Provisions apply to the standard appearing on p. 1423 and for convenience may be cut out and attached to the standard.

[2] This special procedure is set forth in the 1944 A.S.T.M. Year Book.

to read as follows by the addition of the italicized figures and the omission of the figure in brackets:

	Weight per Roll, net average, min., lb.
No bare edge	83.0
2-in. bare edge	81.0
[3] *4*-in. bare edge	*83.0*

Section 10 (a).—Change the first sentence to read as follows by the addition of the italicized figures and the omission of those in brackets:

The roofing shall be put up in rolls of either 108 sq. ft. when made with a 2-in. or no bare-lapping edge, or [111] *114* sq. ft. when made with a [3] *4*-in. bare lapping edge.

Section 12 (a).—Omit the requirements for protective coating on nails prescribed in item (*2*) of this section.

Change the second sentence of item (*3*) of this section to read as follows by the addition of the italicized word and figure and the omission of the figure in brackets:

Where the roofing is intended for application by the [blind] *concealed* nailing method and is provided with a [3] *4*-in. selvage, 150 nails per "square" shall be furnished on a basis of 36-in. roofing.

Section 12 (b).—Change the fifth, sixth, and seventh sentences of item (*2*) of this section to read as follows by the addition of the italicized figures and the omission of those in brackets:

The test specimen shall consist of two strips each 6 in. in length and 1 in. in width cut from the roofing, and one of the strips shall include a [3] *4*-in. bare lapping edge. The specimen shall be prepared for testing by covering the bare lapping edge of the one strip with [1.5] *2* g. of cement, spreading it evenly, and then allowing the strip to remain undisturbed for 10 min. at a temperature of 70 to 90 F. (21 to 32 C.). The reverse side of the other strip shall then be placed upon the cement-coated lapping edge in such a manner as to form a complete specimen [9] *8* in. in length by 1 in. in width that includes a regular [3] *4*-in. cemented lap.

EA - D 607

EMERGENCY ALTERNATE PROVISION
IN
STANDARD SPECIFICATIONS FOR MICA PIGMENT
(D 607 - 42)[1]

ISSUED, SEPTEMBER 16, 1942.

This Emergency Alternate Provision is issued by the American Society for Testing Materials in accordance with a special procedure[2] in the interest of expediting procurement or conservation of materials during the period of National Emergency. It is intended for use where it may be considered by the purchaser of the material as a permissible alternate for the specific application or use desired.

The following Emergency Alternate Provision, when specified, may be used as an alternate in A.S.T.M. Standard Specifications for Mica Pigment (D 607 - 42) and affects only the requirements referred to:

Section 2 (*a*).—Change the requirement for total residue retained on a No. 325 sieve from:

Total residue retained on a No. 325 (44-micron) sieve..... 7.0

to read as follows:

Total residue retained on a No. 325 (44-micron) sieve 10.0

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[1] This Emergency Alternate Provision applies to the standard appearing on p. 835 and for convenience may be cut out and attached to the standard.

[2] This special procedure is set forth in the 1944 A.S.T.M. Year Book.

EA – E 1

EMERGENCY ALTERNATE PROVISIONS
IN
STANDARD SPECIFICATIONS FOR A.S.T.M. THERMOMETERS
(E 1 – 44)[1]

ISSUED, NOVEMBER 9, 1944.

These Emergency Alternate Provisions are issued by the American Society for Testing Materials in accordance with a special procedure[2] in the interest of expediting procurement or conservation of materials during the period of National Emergency. They are intended for use where they may be considered by the purchaser of the material as a permissible alternate for the specific application or use desired.

The following Emergency Alternate Provisions, when specified, may be used as an alternate in A.S.T.M. Standard Specifications for A.S.T.M. Thermometers (E 1 – 44) and affect only the requirements referred to:

A.S.T.M. Low Cloud and Pour Test Thermometers.—To provide thermometers for pour point tests at −65 F. (−54 C.) or lower as covered in the Emergency Alternate Provisions EA–D 97 in A.S.T.M. Standard Method of Test for Cloud and Pour Points (D 97 – 39), change the requirements for these thermometers as follows:

Temperature Range.—In the Centigrade thermometer E1(6C–39), change the lower temperature limit from "−60 C." to read "−80 C."

In the Fahrenheit thermometer E1(6F–39), change the lower temperature limit from "−70 F." to read "−112 F."

Marking on Case.—To agree with the changes in the new ranges of these thermometers, change the required marking on the case of the Centigrade thermometer E1(6C–39) from "−60 C." to read "−80 C. and, on the case of the Fahrenheit thermometer E1(6F–39), from "−70 F." to read "−112 F."

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[1] These Emergency Alternate Provisions apply to the standard appearing on p. 1129 and for convenience may be attached to the standard.

[2] This special procedure is set forth in the 1944 A.S.T.M. Year Book.

A.S.T.M. TENTATIVE STANDARDS RELATING TO NONMETALLIC MATERIALS—CONSTRUCTIONAL

As of November, 1944

(For revisions or change of status in any of these tentative standards, see supplementary publications to be issued in 1945 and 1946.)

Tentative Standards represent the latest thoughts and practices, and are published as tentative by the Society on the recommendations of the committees concerned, prior to adoption as standard.

New and revised Tentative Standards, issued in 1945 and 1946, will appear in the 1945 and 1946 Supplements to the Book of A.S.T.M. Standards. It is important that these Supplements be consulted in case any change of status has occurred with respect to any of these Tentative Standards.

Criticisms of these Tentative Standards should be directed to the Headquarters of the Society, 260 S. Broad St., Philadelphia 2, Pa.

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In the following listing, the tentative standards are grouped according to materials to which they apply.

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[1] See Supplements to be issued in 1945 and 1946 for revision or change of status of any of these tentative standards.

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[2] These specifications were formerly published as Emergency Specifications ES – 8.
[3] These specifications were formerly published as Emergency Specifications ES – 9.
[4] These specifications were formerly published as Emergency Specifications ES – 10.
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Tentative Specifications for

AIR-ENTRAINING PORTLAND CEMENT FOR CONCRETE PAVEMENTS[1]

A.S.T.M. Designation: C 175 – 44 T

ISSUED, 1942; REVISED, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These specifications cover two types of air-entraining portland cement for use in concrete pavements exposed to severe frost action. The two types shall be designated as types IA and IIA and shall correspond respectively to types I and II of the Standard Specifications for Portland Cement (A.S.T.M. Designation: C 150).[3]

Basis of Purchase

2. The purchaser shall specify the type or types desired. When no type is specified, the requirements of type IA shall govern.

Definition

3. For the purpose of these specifications, air-entraining portland cement is the product obtained by pulverizing clinker consisting essentially of hydraulic calcium silicates, to which no additions have been made subsequent to calcination other than water and/or untreated calcium sulfate, except that there shall be interground with the clinker an addition shown to be acceptable, in the amounts indicated, by tests carried out or reviewed by Committee C-1 on Cement[4] (Note).

NOTE.—Tests to determine whether a proposed addition is acceptable will be carried out or reviewed by Committee C-1 on Cement, for those making requests. In the year following that in which an addition has been declared acceptable by the Committee, the name of the addition shall appear as an addendum to Section 3 and any definite limitation on the quantity to be used shall appear in the table of chemical or physical requirements in a revision of the specifications.

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-1 on Cement.

[2] Latest revision accepted by the Society at annual meeting, June, 1944.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

[4] The committee has declared the following additions acceptable:

(*a*) A material known commercially as Vinsol resin, which is manufactured by the Hercules Powder Co. and consists substantially of the petroleum-hydrocarbon insoluble fraction of a coal-tar hydrocarbon extract of pine wood. The Vinsol resin may be added at the mill in the form of an aqueous solution of sodium resinate (the ratio of solid sodium hydroxide to Vinsol resin is 15 parts to 100 parts, by weight).

(*b*) A material known commercially as Darex AEA which is manufactured by the Dewey and Almy Chemical Co. and is substantially a triethanolamine salt of a sulfonated hydrocarbon.

Chemical Requirements

4. Air-entraining portland cements shall conform to the respective chemical requirements prescribed in Table I.

Physical Requirements

5. Air-entraining portland cements shall conform to the respective physical requirements prescribed in Table II.

each shipment, and in a suitable weather-tight building that will protect the cement from dampness and minimize warehouse set.

Inspection

8. Every facility shall be provided the purchaser for careful sampling and inspection, either at the mill or at the site

TABLE I.—CHEMICAL REQUIREMENTS.

	Type IA	Type IIA
Silicon dioxide (SiO_2), min., per cent	...	21.0
Aluminum oxide (Al_2O_3), max., per cent	...	6.0
Ferric oxide (Fe_2O_3), max., per cent	...	6.0
Magnesium oxide (MgO), max., per cent	5.0	5.0
Sulfur trioxide (SO_3), max., per cent	2.0	2.0
Loss on ignition, max., per cent	3.0	3.0
Insoluble residue, max., per cent	0.75	0.75
Ratio of Al_2O_3 to Fe_2O_3	...	0.7 to 2.0
Tricalcium silicate ($3CaO \cdot SiO_2$),[a] max., per cent	...	50
Tricalcium aluminate ($3CaO \cdot Al_2O_3$),[a] max., per cent	...	8
Vinsol resin (methoxyl method), per cent	[b]	[b]
Darex AEA, max., per cent	0.05	0.05

[a] The expressing of chemical limitations by means of calculated assumed compounds does not necessarily mean that the oxides are actually or entirely present as such compounds.

The percentages of tricalcium silicate, dicalcium silicate, and tricalcium aluminate shall be calculated from the chemical analysis as follows:

Tricalcium silicate = (4.07 × per cent CaO) − (7.60 × per cent SiO_2) − (6.72 × per cent Al_2O_3) − (1.43 × per cent Fe_2O_3) − (2.85 × per cent SO_3)

Dicalcium silicate = (2.87 × per cent SiO_2) − (0.754 × per cent $3CaO \cdot SiO_2$)

Tricalcium aluminate = (2.65 × per cent Al_2O_3) − (1.69 × per cent Fe_2O_3)

Oxide determinations calculated to the nearest 0.1 per cent shall be used in the calculations. Compound percentages shall be calculated to the nearest 0.1 per cent and reported to the nearest 1 per cent.

[b] Vinsol resin content is not limited, but is governed by the requirement for air content of mortar prescribed in Table II. The methoxyl test is required to identify the presence of Vinsol resin.

Packaging and Marking

6. When cement is delivered in packages, the name and brand of the manufacturer, the type under these specifications, and the fact that an acceptable interground addition is present, shall be plainly indicated thereon. Similar information shall be provided in the shipping advices accompanying the shipment of packaged or bulk cement. A bag shall contain 94 lb. net. A barrel shall consist of 376 lb. net. All packages shall be in good condition at the time of inspection.

Storage

7. The cement shall be stored in such a manner as to permit easy access for proper inspection and identification of

of the work, as may be specified by the purchaser. The following periods from time of sampling shall be allowed for completion of testing:

3-day test	8 days
7-day test	12 days
28-day test	33 days

Rejection

9. (*a*) The cement may be rejected if it fails to meet any of the requirements of these specifications.

(*b*) Cement remaining in bulk storage at the mill, prior to shipment, for a period greater than 6 months after completion of the tests may be retested and may be rejected if it fails to conform to any of the requirements of these specifications.

(*c*) Packages varying more than 5 per cent from the specified weight may be

TABLE II.—PHYSICAL REQUIREMENTS.

	Type IA	Type IIA
Fineness, specific surface, sq. cm. per g.:		
Average value, *min.*	1600	1700
Minimum value, any one sample	1500	1600
Soundness:		
Autoclave expansion, max., per cent	0.50	0.50
Time of setting (alternate methods):[a]		
Gillmore test:		
Initial set, min., not less than	60	60
Final set, hr., not more than	10	10
Vicat test:		
Initial set, min., not less than	45	45
Final set, hr., not more than	10	10
Air content of mortar, prepared and tested in accordance with Method C 185, per cent by volume	14 ± 4	14 ± 4
Compressive strength, psi.:[b]		
The compressive strength of mortar cubes, composed of 1 part cement and 2.75 parts graded standard sand, by weight, prepared and tested in accordance with Method C 109, shall be equal to or higher than the values specified for the ages indicated below:		
1 day in moist air, 2 days in water	750	600
1 day in moist air, 6 days in water	1500	1250
1 day in moist air, 27 days in water	3000	2500

[a] The purchaser should specify the type of setting time test required. In case he does not so specify, the requirements of the Gillmore test only shall govern.

[b] Unless otherwise specified, compressive strength tests will be made only at 3 and 7 days. The strength at any age shall be higher than the strength at the next preceding age.

rejected; and if the average weight of packages in any shipment, as shown by weighing 50 packages taken at random, is less than that specified, the entire shipment may be rejected.

Methods of Testing

10. The cement shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(*a*) *Sampling.*—Standard Methods of Sampling Hydraulic Cement (A.S.T.M. Designation: C 183).[3]

(*b*) *Chemical Analysis.*—Standard Methods of Chemical Analysis of Portland Cement (A.S.T.M. Designation: C 114 - 44)[3] and Tentative Methods of Chemical Analysis of Portland Cement (A.S.T.M. Designation: C 114 - 44 T).[3]

(*c*) *Fineness.*—Standard Method of Test for Fineness of Portland Cement by the Turbidimeter (A.S.T.M. Designation: C 115).[3]

(*d*) *Autoclave Expansion.*—Standard Method of Test for Autoclave Expansion of Portland Cement (A.S.T.M. Designation: C 151).[3]

(*e*) *Time of Setting.*—Standard Methods of Test for Time of Setting of Hydraulic Cement by the Vicat or Gillmore Needles (A.S.T.M. Designation: C 191).[3]

(*f*) *Air Conient of Mortar.*—Tentative Method of Test for Air Content of Portland-Cement Mortar (A.S.T.M. Designation: C 185).[3]

(*g*) *Compressive Strength.*—Standard Method of Test for Compressive Strength of Hydraulic-Cement Mortars (A.S.T.M. Designation: C 109).[3]

Chemical Requirements

4. Air-entraining portland cements shall conform to the respective chemical requirements prescribed in Table I.

Physical Requirements

5. Air-entraining portland cements shall conform to the respective physical requirements prescribed in Table II.

TABLE I.—CHEMICAL REQUIREMENTS.

	Type IA	Type IIA
Silicon dioxide (SiO_2), min., per cent	...	21.0
Aluminum oxide (Al_2O_3), max., per cent	...	6.0
Ferric oxide (Fe_2O_3), max., per cent	...	6.0
Magnesium oxide (MgO), max., per cent	5.0	5.0
Sulfur trioxide (SO_3), max., per cent	2.0	2.0
Loss on ignition, max., per cent	3.0	3.0
Insoluble residue, max., per cent	0.75	0.75
Ratio of Al_2O_3 to Fe_2O_3	...	0.7 to 2.0
Tricalcium silicate ($3CaO \cdot SiO_2$),[a] max., per cent	...	50
Tricalcium aluminate ($3CaO \cdot Al_2O_3$),[a] max., per cent	...[b]	...[b]
Vinsol resin (methoxyl method), per cent	...	...
Darex AEA, max., per cent	0.05	0.05

[a] The expressing of chemical limitations by means of calculated assumed compounds does not necessarily mean that the oxides are actually or entirely present as such compounds.

The percentages of tricalcium silicate, dicalcium silicate, and tricalcium aluminate shall be calculated from the chemical analysis as follows:

Tricalcium silicate = (4.07 × per cent CaO) − (7.60 × per cent SiO_2) − (6.72 × per cent Al_2O_3) − (1.43 × per cent Fe_2O_3) − (2.85 × per cent SO_3)

Dicalcium silicate = (2.87 × per cent SiO_2) − (0.754 × per cent $3CaO \cdot SiO_2$)

Tricalcium aluminate = (2.65 × per cent Al_2O_3) − (1.69 × per cent Fe_2O_3)

Oxide determinations calculated to the nearest 0.1 per cent shall be used in the calculations. Compound percentages shall be calculated to the nearest 0.1 per cent and reported to the nearest 1 per cent.

[b] Vinsol resin content is not limited, but is governed by the requirement for air content of mortar prescribed in Table II. The methoxyl test is required to identify the presence of Vinsol resin.

Packaging and Marking

6. When cement is delivered in packages, the name and brand of the manufacturer, the type under these specifications, and the fact that an acceptable interground addition is present, shall be plainly indicated thereon. Similar information shall be provided in the shipping advices accompanying the shipment of packaged or bulk cement. A bag shall contain 94 lb. net. A barrel shall consist of 376 lb. net. All packages shall be in good condition at the time of inspection.

Storage

7. The cement shall be stored in such a manner as to permit easy access for proper inspection and identification of each shipment, and in a suitable weathertight building that will protect the cement from dampness and minimize warehouse set.

Inspection

8. Every facility shall be provided the purchaser for careful sampling and inspection, either at the mill or at the site of the work, as may be specified by the purchaser. The following periods from time of sampling shall be allowed for completion of testing:

3-day test	8 days
7-day test	12 days
28-day test	33 days

Rejection

9. (*a*) The cement may be rejected if it fails to meet any of the requirements of these specifications.

(*b*) Cement remaining in bulk storage at the mill, prior to shipment, for a period greater than 6 months after completion of the tests may be retested and may be rejected if it fails to conform to any of the requirements of these specifications.

(*c*) Packages varying more than 5 per cent from the specified weight may be

TABLE II.—PHYSICAL REQUIREMENTS.

	Type IA	Type IIA
Fineness, specific surface, sq. cm. per g.:		
Average value, min.	1600	1700
Minimum value, any one sample	1500	1600
Soundness:		
Autoclave expansion, max., per cent	0.50	0.50
Time of setting (alternate methods):[a]		
Gillmore test:		
Initial set, min., not less than	60	60
Final set, hr., not more than	10	10
Vicat test:		
Initial set, min., not less than	45	45
Final set, hr., not more than	10	10
Air content of mortar, prepared and tested in accordance with Method C 185, per cent by volume	14 ± 4	14 ± 4
Compressive strength, psi.:[b]		
The compressive strength of mortar cubes, composed of 1 part cement and 2.75 parts graded standard sand, by weight, prepared and tested in accordance with Method C 109, shall be equal to or higher than the values specified for the ages indicated below:		
1 day in moist air, 2 days in water	750	600
1 day in moist air, 6 days in water	1500	1250
1 day in moist air, 27 days in water	3000	2500

[a] The purchaser should specify the type of setting time test required. In case he does not so specify, the requirements of the Gillmore test only shall govern.

[b] Unless otherwise specified, compressive strength tests will be made only at 3 and 7 days. The strength at any age shall be higher than the strength at the next preceding age.

rejected; and if the average weight of packages in any shipment, as shown by weighing 50 packages taken at random, is less than that specified, the entire shipment may be rejected.

Methods of Testing

10. The cement shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(*a*) *Sampling.*—Standard Methods of Sampling Hydraulic Cement (A.S.T.M. Designation: C 183).[3]

(*b*) *Chemical Analysis.*—Standard Methods of Chemical Analysis of Portland Cement (A.S.T.M. Designation: C 114 – 44)[3] and Tentative Methods of Chemical Analysis of Portland Cement (A.S.T.M. Designation: C 114 – 44 T).[3]

(*c*) *Fineness.*—Standard Method of Test for Fineness of Portland Cement by the Turbidimeter (A.S.T.M. Designation: C 115).[3]

(*d*) *Autoclave Expansion.*—Standard Method of Test for Autoclave Expansion of Portland Cement (A.S.T.M. Designation: C 151).[3]

(*e*) *Time of Setting.*—Standard Methods of Test for Time of Setting of Hydraulic Cement by the Vicat or Gillmore Needles (A.S.T.M. Designation: C 191).[3]

(*f*) *Air Content of Mortar.*—Tentative Method of Test for Air Content of Portland-Cement Mortar (A.S.T.M. Designation: C 185).[3]

(*g*) *Compressive Strength.*—Standard Method of Test for Compressive Strength of Hydraulic-Cement Mortars (A.S.T.M. Designation: C 109).[3]

Tentative Specifications for

MASONRY CEMENT[1]

A.S.T.M. Designation: C 91 – 44 T

ISSUED, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These specifications cover cement to be used in conjunction with sand in the making of mortar for laying units of masonry construction.

Time of Setting

2. Neat cement paste mixed to normal consistency shall not develop initial set in less than 60 min., as determined by the Gillmore needle. Final set shall be attained within 24 hr.

Soundness

3. A pat of neat cement shall remain firm and hard, and show no signs of distortion, cracking, checking, or disintegration in the steam test for soundness.

Compressive Strength

4. The average compressive strength of 2-in. mortar cubes, made, stored, and tested in accordance with Sections 19 to 29, shall be not less than 400 psi. at the age of 7 days. The average strength at 28 days shall be not less than 750 psi.

Water Retention

5. Cement mortar after suction for 60 sec. shall have a flow greater than 70 per cent of that immediately after mixing, when tested in accordance with the procedure described in Section 30.

Staining

6. This requirement shall apply only when the invitation for bids specifically states that the cement shall be nonstaining to limestone. Nonstaining cement shall contain not more than 0.03 per cent of water-soluble "alkali" when tested in accordance with Section 31.

NOTE.—The amount and nature of the staining material in limestones seem to vary with the stone. The alkali in any cement may, therefore, induce markedly different staining on different stone, even though the stone may have come apparently from the same source. The amount of alkali permitted by the specifications should not cause stain unless stone high in staining material has been used, or unless insufficient means have been used to prevent infiltration of water into the masonry.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-1 on Cement.

[2] Accepted by Committee E-10 on Standards, August 28, 1944.

These specifications are in effect a tentative revision of, and are intended to replace, when adopted, the Standard Specifications for Masonry Cement (C 91 – 40), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Packaging and Marking

7. The cement shall be delivered in suitable bags or barrels with the brand and name of the manufacturer and the net weight plainly marked thereon. All packages shall be in good condition at the time of inspection. Packages varying more than 5 per cent from the weight marked thereon may be rejected, and if the average weight of packages in any shipment as shown by weighing 50 packages taken at random is less than the weight marked thereon, the entire lot represented may be rejected.

Storage

8. The cement shall be stored in a suitable weathertight building that will protect it from dampness, and in such a manner as to permit safe and easy access for proper inspection and identification of each lot.

Inspection

9. Every facility shall be provided the purchaser for the necessary inspection and sampling.

Rejection

10. The cement shall be rejected if it fails to meet any of the requirements of these specifications. Cement remaining in storage prior to shipment for a period greater than 6 months after test shall be retested, and shall be rejected if it fails to meet any of the requirements of these specifications.

SAMPLING AND METHODS OF TESTING

Size and Number of Samples

11. Each sample of cement selected for purpose of tests shall weigh at least 5 lb. and shall represent not more than 1200 bags. If only one sample is taken, it shall weigh at least 10 lb.

Shipment of Samples

12. Samples shall be shipped and stored in airtight, moistureproof containers.

Temperature

13. The temperature of the room and dry materials shall be maintained at not less than 20 C. (68 F.) and not more than 27.5 C. (81.5 F.). The temperature of the mixing water, moist closet, and water in the cube storage tank shall not vary from 21 C. (70 F.) by more than 1.7 C. (3 F.).

Preparation of Samples

14. The sample of cement shall be given a preliminary sieving by being passed through a No. 20 (840-micron) sieve[3] in order to thoroughly mix the sample, break up lumps, and remove foreign materials.

Preparation of Cement Pastes

15. A 500-g. portion of the cement shall be placed in the form of a crater on a plane, nonabsorbent plate. About one half of the mixing water necessary for normal consistency (Section 16) shall be poured into the crater, the cement mixed into the water with the finger tips until all the water is taken up by the cement, the remainder of the water added, and the paste then mixed with the fingers until all the water is absorbed. The mixing operation shall then be completed by continuously and vigorously squeezing and kneading the paste with the hands for at least 3 min. During the operation of mixing, the hands shall be protected by rubber gloves.

Normal Consistency

16. Normal consistency shall be determined by the Vicat apparatus in

[3] Detailed requirements for these sieves are given in the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

accordance with the Standard Method of Test for Normal Consistency of Hydraulic Cement (A.S.T.M. Designation: C 187),[4] except that the mixing of the paste shall be performed in accordance with Section 15 of these specifications.

Time of Setting Test

17. The time of setting shall be determined by the Gillmore needle method in accordance with the Standard Methods of Test for Time of Setting of Hydraulic Cement by the Vicat or Gillmore Needles (A.S.T.M. Designation: C 191),[4] except that the mixing of the paste shall be performed in accordance with Section 15 of these specifications.

Soundness Test

18. Pats of neat cement, made and tested in accordance with the Standard Method of Test for Soundness of Hydraulic Cement over Boiling Water (Pat Test) (A.S.T.M. Designation: C 189),[4] except that the pastes shall be prepared in accordance with Section 15 of these specifications, shall remain firm and hard and show no signs of distortion, checking, cracking, or disintegration.

Compressive Strength Test

19. *Apparatus:* (*a*) *Scales.*—The scales used in weighing materials for mortar mixes shall conform to the following requirements: On scales in use the permissible variation at a load of 2000 g. shall be plus or minus 2.0 g. The permissible variation on new scales shall be one half of this value. The sensibility reciprocal[5] shall be not greater than twice the permissible variation.

(*b*) *Weights.*—The permissible variations on weights in use in weighing materials for mortar mixes shall be as prescribed in Table I. The permissible variations on new weights shall be one half of the values in Table I.

TABLE I.—PERMISSIBLE VARIATIONS ON WEIGHTS.

Weight, g.	Permissible Variations on Weights in Use, plus or minus, g.
1000	0.5
900	0.45
750	0.4
500	0.35
300	0.3
250	0.25
200	0.20
100	0.15
50	0.10
20	0.05
10	0.04
5	0.03
2	0.02
1	0.01

(*c*) *Sieves.*—Square-hole, woven wire cloth No. 20 (840-micron), and No. 30 (590-micron) sieves conforming to the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11)[4] shall be used.

(*d*) *Glass Graduates.*—Glass graduates of suitable capacities (preferably large enough to measure the mixing water in a single operation) shall be made to deliver the indicated volume at 20 C. (68 F.). The permissible variation shall be plus or minus 2 ml. These graduates shall be subdivided to at least 5 ml. The main graduation lines shall be circles and shall be numbered. The least graduations shall extend at least one seventh of the way around, and intermediate graduations shall extend at least one fifth of the way around.

(*e*) *Specimen Molds.*—Molds for the 2-in. cube test specimens shall be tight fitting. The parts of the molds when assembled shall be positively held together. The molds shall be made of hard

[4] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

[5] The sensibility reciprocal is a measure of the sensitiveness of a balance, and is the weight required to move the position of equilibrium of the pointer one division. For a complete definition of sensibility reciprocal, see "Specifications, Tolerances, and Regulations for Commercial Weighing and Measuring Devices," *Handbook H29*, Nat. Bureau Standards, September, 1942, pp. 87 and 88.

metal not attacked by the cement mortar. For new molds the Rockwell hardness number of the metal shall be not less than B 55. There shall be sufficient material in the sides of the molds to prevent spreading or warping. The interior faces of the molds shall be plane surfaces with a permissible variation of 0.001 in. for new molds and 0.002 in. for molds in use. The distances between opposite faces shall be 2 ± 0.005 in. for new molds, and 2 ± 0.02 in. for molds in use. The height of the molds, measured separately for each cube compartment, shall be 2 in. with permissible variations of plus 0.01 in. and minus 0.005 in. for new molds, and plus 0.01 in. and minus 0.015 in. for molds in use. The angle between adjacent interior faces, and between interior faces and top and bottom planes of the mold shall be 90 ± 0.5 deg. measured at points slightly removed from the intersection of the faces.

(*f*) *Mixing Bowl.*—The mixing bowl shall be of enamelware or other nonabsorbent, noncorroding material and shall have a capacity of about 1 gal.

(*g*) *Flow Table.*—The flow table apparatus shall consist of a rigid metal frame and a circular rigid table 10 in. in diameter with a shaft attached perpendicular to the table top. The table with attached shaft shall be mounted on the frame in such a manner that it can be raised and dropped vertically through a fixed height of ½ in. by means of a rotated cam. The table top shall have a plane surface and be of noncorrodible metal. The table and the attached shaft shall weigh 9 ± 0.1 lb. The end of the shaft shall not strike upon the cam at the end of the drop. The surfaces of the table and the frame which come into contact at the end of the drop shall be plane and parallel with the upper surface of the table and the material of these parts shall be hard metal to prevent cushioning effect. The contact faces of the cam and the shaft shall be such that the table does not rotate more than one revolution during the 25 drops. The frame shall be attached rigidly to a concrete pedestal which shall be attached rigidly to the floor. The concrete pedestal shall be at least 8 in. square or in diameter at the top and at least 25 in. in height with a base suitable for rigid attachment to the floor and shall weigh at least 100 lb. The table top, after the frame has been mounted on the pedestal, shall be level along any two diameters at right angles to each other in both the raised and the lowered position.

(*h*) *Flow Mold.*—The flow mold shall be made of noncorrodible material and shall be 4 in. in inside diameter at the base, 2.75 in. in inside diameter at the top, and 2 in. in height.

(*i*) *Trowel.*—The trowel shall have a steel blade 4 to 6 in. in length, with straight edges.

(*j*) *Testing Machine.*—The testing machine may be either the hydraulic or the screw-type with sufficient opening between the upper bearing surface and the lower bearing surface of the machine to permit the use of verifying apparatus. The load applied to the test specimen shall be indicated with an accuracy of plus or minus 1.0 per cent. The upper bearing shall be a spherically seated, hardened metal block firmly attached at the center of the upper head of the machine. The center of the sphere shall lie at the center of the surface of the block in contact with the specimen. The block shall be closely held in its spherical seat, but shall be free to turn in any direction. The diagonal or diameter[6] of the bearing surface shall be only slightly greater

[6] A diameter of 3⅛ in., which is large enough for testing 3 by 6-in. cylinders, is satisfactory provided that the lower bearing block has a diameter slightly greater than the diagonal of the face of the 2-in. cube but not more than 2.9 in., and is centered with respect to the upper bearing block and held in position by suitable means.

than the diagonal of the face of the 2-in. cube in order to facilitate accurate centering of the specimen. A hardened metal bearing block shall be used beneath the specimen to minimize wear of the lower platen of the machine. The bearing block surfaces intended for contact with the specimen shall have a Rockwell hardness number not less than C 60. These surfaces shall not depart from plane surfaces by more than 0.0005 in. when the blocks are new and shall be maintained within a permissible variation of 0.001 in.

20. *Standard Sand.*—The sand used for making test specimens shall be natural silica sand from Ottawa, Ill., graded to pass a No. 20 (840-micron) sieve and retained on a No. 30 (590-micron) sieve.[3] This sand shall be considered standard when not more than 15 g. are retained on the No. 20 sieve, and not more than 5 g. pass the No. 30 sieve after 5 min. of continuous sieving of a 100-g. sample in the manner specified for sieving cement in the Standard Method of Test for Fineness of Hydraulic Cement by the No. 200 Sieve (A.S.T.M. Designation: C 184).[4]

21. *Number of Specimens.*—Three or more specimens shall be made for each period of test specified.

22. *Preparing Specimen Molds.*—The interior faces of the specimen molds shall be thinly covered with mineral oil or light cup grease. After assembling the molds, excessive oil or grease shall be removed from the interior faces and the top and bottom surfaces of each mold. Molds shall then be set on plane, nonabsorbent base plates which have been thinly coated with mineral oil, petrolatum, or light cup grease.

23. (*a*) *Proportions for Standard Mortar.*—The mortar shall consist of 1 part cement to 3 parts of standard sand by weight. The quantities of dry materials to be mixed at one time shall be 500 g. of cement and 1500 g. of sand. The quantity of mixing water, measured in milliliters, shall be such as to produce a flow of between 100 and 115 when determined in accordance with Section 24.

(*b*) *Mixing of Mortars.*—The mortars shall be mixed in a nonabsorbent bowl of about 1-gal. capacity. A measured quantity of water shall be poured into the bowl which has previously been wiped with a damp cloth. The cement shall then be added and stirred into the water with the fingers of one hand until all the cement is wetted. Approximately 800 g. of sand shall then be added and the stirring continued for 30 sec. The remainder of the 1500 g. of sand shall then be added and the mortar mixed for 75 sec. by vigorous and continued stirring, squeezing, and kneading with one hand. The mortar shall then be allowed to stand for 60 sec. and then mixed for another 60 sec. During the operation of mixing, the hands shall be protected by rubber gloves.

24. *Determination of Flow.*—The flow-table top shall be carefully wiped dry and the flow mold placed at the center. Immediately after completing the mixing operation, the mold shall be filled with mortar gently pressed into place by the finger tips to insure uniform filling. The mortar shall then be cut off to a plane surface, flush with the top of the mold, by drawing the straight edge of a trowel (held nearly perpendicular to the mold) with a sawing motion across the top of the mold. The mold shall be lifted away from the mortar 30 sec. after completing the mixing operation. Immediately the table shall be dropped through a height of $\frac{1}{2}$ in., 25 times in 15 sec. The flow is the resulting increase in diameter of the mortar mass, expressed as a percentage of the original diameter. Trial mortars shall be made with varying percentages of water until the specified flow is ob-

tained. Each trial shall be made with fresh mortar.

25. *Molding Test Specimens.*—Immediately after mixing the standard mortar in accordance with the procedure described in Section 23 (*b*), the mortar shall be placed in the specimen molds, whichs hall rest on plane, nonabsorbent plates. The molds shall be half filled with mortar, the mortar puddled into place with the finger tips of the gloved hand, the mold then filled to overflowing, and the mortar again puddled with the finger tips. The mortar shall then be troweled off flush with the top of the molds.

26. *Storage of Test Specimens.*—All test specimens, immediately after molding, shall be kept in the molds on plane plates in a damp closet, maintained at a relative humidity of 90 per cent or more, for from 48 to 52 hr. in such a manner that the upper surfaces shall be exposed to the moist air. The cubes shall then be removed from the molds and placed in the damp closet for 5 days in such a manner as to allow free circulation of air around at least five faces of the specimens. At the age of 7 days the cubes for the 28-day tests shall be immersed in clean running water in storage tanks of noncorrodible materials.

27. *Testing of Specimens.*—Testing of the cube specimens shall be carried out immediately upon their removal from the moist closet for 7-day specimens, and from storage water for all other specimens. If more than one specimen at a time is removed from the storage water for testing, these cubes shall be placed in a pan of water of sufficient depth to completely immerse each cube until its removal for testing. The load shall be applied to faces of the cubes that were in contact with the true plane surfaces of the mold. These faces shall be checked by application of an accurate straightedge. If appreciable curvature is present, the face or faces shall be ground to a plane surface before loading, or the specimens shall be discarded. Loose sand grains or incrustations shall be removed from the contact faces, and the cubes shall then be carefully placed in the testing machine below the center of the upper bearing block. No cushioning or bedding materials shall be used. The loading up to 25 per cent of the expected maximum load may be applied at any convenient rate, after which the specimens shall be loaded continuously to failure at a rate or rates which shall at no time be less than 1000 nor more than 6000 psi. per min.

28. *Calculations.*—The total maximum load indicated by the testing machine shall be recorded, and the compressive strength calculated in pounds per square inch from the cross-sectional area of the cube tested.

29. *Faulty Cubes and Retests.*—Cubes that are manifestly faulty or that give strengths differing by more than 10 per cent from the average value of all test specimens made from the same sample and tested at the same period shall not be considered in determining the compressive strength. After discarding cubes or strength values, if less than two strength values are left for determining the compressive strength at any given period, a retest shall be made.

Water Retention Test

30. (*a*) *Apparatus.*—For the water retention test, an apparatus essentially the same as that shown in Fig. 1 shall be used. This apparatus consists of a water aspirator controlled by a mercury column relief and connected by way of a three-way stopcock to a funnel upon which rests a perforated dish. A mercury manometer, connected as shown in Fig. 1, indicates the vacuum. A rubber gasket shall be sealed to the top of the

funnel and shall be kept wet during a test to insure a seal between the funnel and dish. The perforated dish shall be made of nonabsorbent material. Hardened filter paper equivalent to Carl Schleicher and Schull filter paper No. 575 shall be used. It shall be of such diameter that it will lie flat and completely cover the bottom of the dish.

(*b*) *Method.*—The mortar shall be mixed to a flow of from 100 to 115 per cent in accordance with the procedure described in Section 23(*b*). Immediately after making the flow test, the mortar on the flow table shall be remixed for 30 sec. with that remaining in the mixing bowl. Immediately after remixing, the mortar shall be uniformly distributed, without compacting, over the sheet of dampened filter paper in the perforated dish and the surface leveled off flush with the rim of the dish by drawing a straightedge across the dish with a slightly sawing motion. The dish shall then be seated on the wetted gasket, and with the mercury relief column previously adjusted so as to maintain a vacuum of 2 in., the stopcock shall be turned to apply the vacuum to the funnel. After suction for 60 sec., the stopcock shall be quickly turned to expose the funnel to atmos-

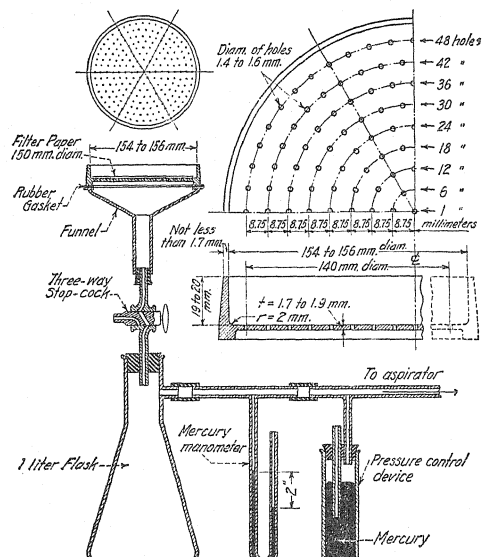


FIG. 1.—Apparatus Assembly for the Water Retention Test.

pheric pressure. The contents of the dish shall then be immediately removed by means of a putty knife or square-end spatula and placed in the mold on the flow table. As each portion of mortar is placed in the mold it shall be well puddled with glove-covered fingers. When the mold is filled, the mortar shall be smoothed off level with the top of the mold, using the edge of a trowel, and the flow determined as previously. The entire operation shall be carried out without interruption and as quickly as possible and shall require not more than 7 min. for completion, starting from the completion of the mixing of the mortar for the first flow determination. Both flow determinations shall be made in accordance with the procedure described in Section 24, special care being taken to fill the mold uniformly when obtaining the flow after suction.

Staining Test (Determination of Water-Soluble Alkali)

31. Water-soluble alkali shall be determined in accordance with Section 20 of the Standard Methods of Chemical Analysis of Portland Cement (A.S.T.M. Designation: C 114).[4]

Tentative Methods of

CHEMICAL ANALYSIS OF PORTLAND CEMENT[1]

A.S.T.M. Designation: C 114 – 44 T

ISSUED, 1937; REVISED, 1938, 1939, 1940, 1942, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

General Requirements

1. The sampling, apparatus, purity of water and reagents, concentration of reagents, glassware, and number of chemical determinations shall conform to the requirements prescribed in Sections 2 to 7 (*a*) of the Standard Methods of Chemical Analysis of Portland Cement (A.S.T.M. Designation: C 114 – 44).[3]

SODIUM OXIDE AND POTASSIUM OXIDE[4]

Rapid Alternate Method

NOTE.—A determination of sodium oxide and potassium oxide in accordance with Sections 18 and 19 of the Standard Methods of Chemical Analysis of Portland Cement (A.S.T.M. Designation: C 114 – 44) of the American Society for Testing Materials[3] requires approximately three days. A determination by this alternate method requires approximately two days. The standard method is more suitable for an occasional analysis. The alternate method may be used when a large number of determinations are made or when such a determination is made frequently. The method may present a number of difficulties to an inexperienced operator, but they can be overcome with experience. In case of dispute, the standard method should be used.

Reagents

2. (*a*) *Zinc Uranyl Acetate Reagent.*—Shake a mixture of 20 g. of uranyl acetate ($UO_2(CH_3CO_2)_2 \cdot 2H_2O$), 60 g. of zinc acetate ($Zn(CH_3CO_2)_2 \cdot 2H_2O$), 5 ml. of glacial acetic acid, and 175 ml. of warm water continuously for about 1 hr. or at intervals for a few hours. If the solution is clear, add a little NaCl or sodium zinc uranyl acetate ($NaZn(UO_2)_3(CH_3CO_2)_9 \cdot 6H_2O$) to the solution and shake it again. It is essential that the reagent be saturated with respect to sodium zinc uranyl acetate at the temperature at which the

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee C-1 on Cement.

[2] These methods when adopted as standard are intended to be incorporated in the Standard Methods of Chemical Analysis of Portland Cement (A.S.T.M. Designation: C 114 – 44), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

[4] This procedure is chiefly a modification of a method described by Francis W. Glaze, of the National Bureau of Standards, in a paper entitled "On the Direct Determination of Soda in Soda-Lime Glasses by Precipitation as Uranyl Zinc Sodium Acetate," *Journal*, Am. Ceramic Soc. Vol. 14, No. 6, June, 1931, p. 450. The use of uranyl zinc acetate to precipitate sodium was proposed by H. H. Barber and I. M. Kolthoff, "A Specific Reagent for the Rapid Gravimetric Determination of Sodium," *Journal*, Am. Chemical Soc., Vol. 50, p. 1625 (1928). The washing of potassium chloroplatinate with Gladding wash and its direct weighing were suggested by Fred P. Diener of Universal Atlas Cement Co. Many minor suggestions were contributed by a number of analysts.

reagent is used. Allow the reagent to stand until its temperature is constant. Filter small portions of it as required, keeping the temperature as uniform as practicable in all operations involving the preparation of the reagent and alcoholic wash and the precipitation, filtration, and washing of the sodium zinc uranyl acetate.

(*b*) *Alcoholic Wash.*—Add some sodium zinc uranyl acetate to ethanol which contains acetic acid (1 per cent), and shake the solution continuously for about 1 hr. or at intervals for a few hours. Filter portions of the wash as required. (See Paragraph (*a*) in regard to temperature.) The ethanol may be absolute or "95 per cent" and may be pure or denatured according to Formula No. 2b or 3a of the U. S. Bureau of Internal Revenue.

(*c*) *Chloroplatinic Acid.*—Dissolve 5 g. of $H_2PtCl_6 \cdot 6H_2O$ in 95 ml. of water and 5 ml. of HCl (sp. gr. 1.18).

(*d*) *Gladding Wash.*—Dissolve 100 g. of NH_4Cl in 500 ml. of water and add to the solution an amount of K_2PtCl_6 that is more than sufficient to make a saturated solution (about 1 g.). Shake the mixture frequently for 6 to 8 hr., allow it to stand at least 12 hr., and filter. The residue may be used for the preparation of a new supply of the wash.

Procedure

3. (*a*) *Preparation of Solution.*—To 2 g. of cement in a platinum dish, preferably of the flat-bottom type, add 5 ml. of cool water, stir the mixture with a platinum rod, add carefully 10 ml. of diluted HNO_3 (1:1), and continue the stirring until the cement is completely decomposed. Add 10 ml. of $HClO_4$ (70 per cent) and 25 ml. of HF (48 per cent) (Note 1) to the solution and mix it thoroughly with the rod. Remove the rod and rinse it with water. Evaporate the solution to dryness or fumes of $HClO_4$ (Note 2). Cool the dish, wash down the side with a little hot water, and repeat the evaporation. Drive off the excess $HClO_4$ by cautiously moving a flame under the dish or increasing the temperature of a radiator, taking care not to heat the dish to redness. Cover the dish with a watch glass of fused silica or heat-resistant glass, and convert the perchlorates to chlorides by using stronger heat but still avoiding red heat. The residue fuses to a brown mass and may glow from an exothermic reaction. The reaction may be carried out by heating a small part of the residue at a time and applying no more heat than necessary to keep the reaction going. After the completion of the reaction, continue the heating for 1 or 2 min. below red heat. Cool the dish and watch glass (Note 3). Rinse the watch glass into a beaker. Add a little water to the dish, let the residue stand a few minutes to soften, and grind it with a glass mushroom or pestle to a smooth paste (Note 4). Wash the contents into the beaker and dilute them to 40 to 50 ml. Boil the contents for 15 min., keeping the volume between 25 and 40 ml. Add a little macerated filter pulp to the contents, and filter the solution through a filter paper of medium or fine porosity into a 100-ml. volumetric flask which contains sufficient HCl to acidify the filtrate (Note 5). Wash the filter paper and residue at least ten times until the volume is nearly 100 ml. Cool the filtrate (Note 6), dilute to the mark, and mix thoroughly.

NOTE 1.—Care should be taken that the acid does not carry any wax from its wax bottle, as organic matter in the presence of $HClO_4$ at a high temperature may cause an explosion.

NOTE 2.—The evaporation requires care as the contents have a tendency to bump or spatter, especially when the dish is in contact with a flame or a hot plate at a high temperature. The best way to handle the evaporation depends on the equipment available to the operator and the

organization of his work. If practicable, the dish may be left on a water bath or a hot plate at a low temperature overnight. If the hot plate is too hot, a mat of asbestos may be used under the dish. A radiator may be used in which the solution is heated by infrared reflector drying lamps placed below or above the dish, or both. If a gas-fired radiator is used, its bottom should be closed so that the dish is heated by radiation rather than by conduction. A radiator of this type may be constructed out of stock 2-in. plumbing fittings: Place a 3-in. support ring over a close nipple and screw a cap and coupling on the nipple until they come together and hold the ring firmly. As the iron is thick, a burner of the Meker type may be used for heating. If a similar radiator is made of thin copper or galvanized iron, an ordinary bunsen burner may be used. The contact of platinum with iron should be avoided. If the residue is allowed to stand overnight after the second evaporation, it may be placed in an oven or desiccator to prevent delay the next day due to absorption of moisture.

NOTE 3.—As ammonium chloroplatinate is like potassium chloroplatinate in being insoluble in ethanol, all following steps involving the determination of potassium oxide should be carried out in an atmosphere free of ammonia and ammonium salts.

NOTE 4.—The residue should be thoroughly ground to insure the complete extraction of sodium and potassium chlorides. If the bottom of the dish is round and the grinding cannot be done satisfactorily, the residue may be transferred to a casserole with the aid of water and a rubber policeman. Decant most of the water into the beaker and grind the residue.

NOTE 5.—One or two drops of acid are usually sufficient. Filtration under suction may be employed. A convenient arrangement is to use a large bell glass, the edge of which is greased and in contact with a piece of plate glass. The top should have a 1- to 2-in. opening fitted with a two-hole rubber stopper. Insert a funnel and a glass tubing through the stopper. Connect the tubing with a T-shape connecting tube and an aspirator or vacuum pump. The connecting tube enables one to release the vacuum without turning the aspirator or pump off. The end of the tubing within the bell glass should be curved toward the side and fitted with a rubber tubing which extends to the bottom of the bell glass. This prevents the spoilage of work by the back rush of air or water. The arrangement makes it possible to filter directly into vessels of many types and sizes with suction.

NOTE 6.—It is not necessary to cool to any particular temperature so long as the flask and pipettes used in the following steps are calibrated at the same temperature and the temperature of the solution is close to the temperature of calibration.

Determination of Na_2O

(*b*) Transfer a 20-ml. aliquot of the solution of the sample (Paragraph (*a*)) to a small vessel and evaporate it to dryness (Note 7). Cool the vessel, take the residue up in 1 ml. of water, add 15 ml. of the zinc uranyl acetate reagent while stirring, and allow the contents to stand for 30 to 60 min. (Note 8) with frequent stirring. Filter the solution through a weighed (Note 9), 15-ml., fritted glass filtering crucible of medium porosity, or a Gooch crucible, with suction. Transfer the precipitate to the crucible and scrub and rinse the beaker with a rubber policeman, using small quantities of the zinc uranyl acetate reagent. Wash the precipitate six times with 2- to 3-ml. portions of the alcoholic wash (Note 10), being careful to wash down the side of the crucible, and three times with 4- to 5-ml. portions of anhydrous ether. Draw air through the crucible until the odor of ether disappears, wipe the outside of the crucible with a damp cloth, place the crucible in a desiccator for 1 hr. (or 15 min. if *in vacuo*), and weigh the sodium zinc uranyl acetate ($NaZn(UO_2)_3(CH_3CO_2)_9 \cdot 6H_2O$).

(*c*) *Blank.*—Make a blank determination, following the same procedure and using the same amounts of reagents, and correct the results obtained in the analysis accordingly.

(*d*) *Calculation.*—Calculate the percentage of Na_2O as follows:

$$Na_2O, \text{ per cent} = W \times 5.0375$$

where:

W = grams of $NaZn(UO_2)_3(CH_3CO_2)_9 \cdot 6H_2O$, and

5.0375 = molecular ratio of Na_2O to 2 $NaZn(UO_2)_3(CH_3CO_2)_9 \cdot 6H_2O$

(0.02015) divided by the weight of sample in the aliquot used (0.4 g.) and multiplied by 100.

NOTE 7.—The first part of the evaporation may be done over a burner or hot plate with a stream of air directed on the surface and finished on a water bath or in a drying oven at 105 C.

NOTE 8.—The solution should stand at least 1 hr. when the content of Na_2O is low (less than 0.2 per cent).

NOTE 9.—Wash, dry, and weigh the empty crucible just as it is done when filtered sodium zinc uranyl acetate is present.

NOTE 10.—When the reagent and alcoholic wash are mixed and allowed to stand, a white precipitate results which does not dissolve in an excess of the wash. The washing with the alcoholic wash should be done rapidly.

Determination of K_2O

(*e*) Transfer a 75-ml. aliquot of the solution of the sample (Paragraph (*a*)) to a vessel and evaporate the solution to about 5 ml. Add 5 ml. of the chloroplatinic acid to the solution and continue the evaporation on a water bath until the liquid solidifies to a soft mass upon cooling (Note 11). Add 30 ml. of ethanol (85 per cent) to the vessel and grind the residue finely with a glass mushroom. Allow the contents to stand for about 1 hr. with frequent stirring and filter through a weighed, 15-ml., fritted glass filtering crucible of medium porosity with suction. Scrub and rinse the vessel with as little ethanol (85 per cent) as possible. Wash the crucible and residue five times with 3- to 5-ml. portions of ethanol (85 per cent), then six times with the Gladding wash with the suction being shut off and the residue churned up thoroughly with a fine stream of the wash each time (Note 12), and finally six times with ethanol (85 per cent) with care taken to wash down the side of the crucible. Dry the crucible at 105 to 110 C. for at least 2 hr. or 130 to 135 C. for at least 30 min., cool in a desiccator, and weigh the K_2PtCl_6.

(*f*) *Blank.*—Make a blank determination, following the same procedure and using the same amounts of reagents, and correct the results obtained in the analysis accordingly.

(*g*) *Calculation.*—Calculate the percentage of K_2O as follows:

$$K_2O, \text{ per cent} = W \times 12.92$$

where:

W = grams of K_2PtCl_6, and
12.92 = molecular ratio of K_2O to K_2PtCl_6 (0.1938) divided by the weight of sample in the aliquot used (1.5 g.) and multiplied by 100.

NOTE 11.—The content of K_2O is assumed to be less than 1.5 per cent. Five milliliters of the chloroplatinic acid is about twice the amount necessary to precipitate the potassium in cement having a K_2O content of 1.5 per cent. If the content is much greater than 1.5 per cent, the amount of the acid should be increased in proportion. The liquid may contain so much calcium chloride that toward the end of the evaporation most of the calcium chloride and some of the K_2PtCl_6 may crystallize out, giving slush instead of a syrupy solution. The last stage of the evaporation requires care, and it may be necessary to cool and re-evaporate a few times. If the evaporation is prolonged too long, the chloroplatinic acid may be partially decomposed or the solidified mass may be hard to break up in ethanol.

NOTE 12.—The K_2PtCl_6 is contaminated with considerable material which is insoluble in ethanol but soluble in the Gladding wash. It is thus essential that the washing with the Gladding wash be done thoroughly.

SULFIDE SULFUR[5]

Apparatus

4. *Gas Generating Flask.*—Connect a dry 500-ml. boiling flask with a long-stem separatory funnel and a small connecting bulb by means of a rubber stopper. Bend the stem of the funnel

[5] This procedure is based on National Bureau of Standards *Research Paper 968*, by Harry A. Bright, "Determination of Sulphur Occurring as Sulphide in Portland Cement," *Journal of Research*, Nat. Bureau Standards, Vol. 18, No. 2, February, 1937, p. 137.

so that it will not interfere with the connecting bulb, adjust the stem so that the lower end is close to the bottom of the flask, and connect the opening of the funnel with a source of compressed air. Connect the bulb with an L-shaped glass tube and a straight glass tube about 20 cm. in length. Insert the straight glass tube in a tall-form, 400-ml. beaker. A three-neck Woulff bottle with a long glass tubing in the middle opening, placed between the source of compressed air and the funnel, is a convenient aid in the regulation of the air flow. If the air contains H_2S or SO_2, a solution of lead acetate or some other suitable absorbent shall be used in the bottle. Rubber used in the apparatus shall be of a pure-gum grade low in sulfur and shall be cleaned with warm HCl.

Reagents

5. (*a*) *Starch Solution.*—To 100 ml. of boiling water, add a cool suspension of 1 g. of soluble starch in 5 ml. of water and cool. Add a cool solution of 1 g. of NaOH in 10 ml. of water, then 3 g. of KI, and mix thoroughly.

(*b*) *Potassium Permanganate Solution (approximately 0.03 N).*—Prepare a solution of $KMnO_4$ on the basis of 0.94 g. per liter. The solution should not be filtered through any filter containing organic matter. It is most convenient to siphon off clear solution without disturbing the sediment on the bottom of the bottle. Standardize the solution against about 0.15 g. of sodium oxalate oxidimetric standard furnished by the National Bureau of Standards (standard sample No. 40) according to the directions furnished with the sodium oxalate.

(*c*) *Sodium Thiosulfate Solution (approximately 0.03 N).*—Prepare a solution of $Na_2S_2O_3$ on the basis of 7.4 g. of $Na_2S_2O_3 \cdot 5H_2O$ per liter.

(*d*) *Potassium Iodate Solution (approximately 0.03 N).*—Prepare a solution of KIO_3 and KI on the basis of 1.12 g. of KIO_3 and 12 g. of KI per liter. Standardize the solution as follows: To a cool solution of 1 g. of KI in 300 ml. of water and 10 ml. of HCl (sp. gr. 1.18) in a 500-ml. flask, add about 25 ml. of the $KMnO_4$ solution, swirl the solution gently, stopper the flask, and let stand for 5 min. Titrate the liberated iodine with the thiosulfate solution until the color nearly fades. Add 2 ml. of the starch solution, continue the titration until the blue color is destroyed, and back-titrate with the $KMnO_4$ solution (Paragraph (*b*)) until the blue color just reappears. Repeat the titration with the KIO_3 solution substituted for the $KMnO_4$ solution. Calculate the sulfur equivalent of the standard solution of KIO_3 in grams per milliliter as follows:

$$E = \frac{A \times C \times G \times 0.2392}{B \times D \times F}$$

where:

E = sulfur equivalent of the KIO_3 solution in grams per milliliter,
A = grams of $Na_2C_2O_4$ used in the standardization of the $KMnO_4$ solution,
B = milliliters of $KMnO_4$ solution required by A,
C = milliliters of $KMnO_4$ solution used in the standardization of the KIO_3 solution,
D = milliliters of $Na_2S_2O_3$ solution required by C,
F = milliliters of KIO_3 solution used in the standardization of the KIO_3 solution, and
G = milliliters of $Na_2S_2O_3$ solution required by F.

NOTE.—A normal solution of $KMnO_4$ or KIO_3 is equivalent to 0.06701 g. of $Na_2C_2O_4$ or 0.01603 g. of sulfur. The number 0.2392 is obtained by dividing 0.01603 by 0.06701. The KIO_3 and $KMnO_4$ solutions should be standardized frequently, but, as the $Na_2S_2O_3$ solution

is more stable, the KIO_3 solution may sometimes be standardized against the $Na_2S_2O_3$ solution alone and without the last values of *A*,*B*,*C*, and *D* being changed.

(*e*) *Stannous Chloride Solution.*—To 10 g. of $SnCl_2 \cdot 2H_2O$ in a small flask, add 7 ml. of diluted HCl (1:1), warm the mixture gently until the salt is dissolved, cool the solution, and add 95 ml. of water. This solution should be prepared as needed, as the salt tends to hydrolyze.

(*f*) *Ammoniacal Zinc Sulfate Solution.*—Dissolve 50 g. of $ZnSO_4 \cdot 7H_2O$ in 150 ml. of water and 350 ml. of NH_4OH (sp. gr. 0.90). Filter the solution after allowing it to stand at least 24 hr.

(*g*) *Ammoniacal Cadmium Chloride Solution.*—Dissolve 15 g. of $CdCl_2 \cdot 2H_2O$ in 150 ml. of water and 350 ml. of NH_4OH (sp. gr. 0.90). Filter the solution after allowing it to stand at least 24 hr.

Procedure

6. (*a*) Place 15 ml. of the ammoniacal $ZnSO_4$ solution (Note 1) and 285 ml. of water in the beaker. Put 5 g. of the sample (Note 2) and 10 ml. of water in the flask and shake the flask gently to wet and disperse the cement completely. This step and the following one should be performed rapidly to prevent the setting of the cement. Connect the flask with the funnel and bulb. Add 25 ml. of the $SnCl_2$ solution through the funnel and shake the flask. Add 100 ml. of diluted HCl (1:3) through the funnel and shake the flask. During these shakings, keep the funnel closed and the delivery tube in the ammoniacal $ZnSO_4$ solution. Connect the funnel with the source of compressed air, open the funnel, start a slow stream of air, and heat the flask and contents slowly to boiling. Continue the boiling gently for 5 or 6 min., cut off the heat, and continue the passage of air for 3 or 4 min. Disconnect the delivery tube and leave it in the solution for use as a stirrer. Cool the solution to 20 to 30 C. (Note 3) add 2 ml. of the starch solution and 40 ml. of diluted HCl (1:1), and titrate immediately with the KIO_3 solution until a permanent blue color is obtained (Note 4).

(*b*) *Blank.*—Make a blank determination, following the same procedure and using the same amounts of reagents, and correct the results obtained in the analysis accordingly.

(*c*) *Calculation.*—Calculate the percentage of sulfur (Note 5) as follows:

$$\text{Sulfur, per cent} = EV \times 20$$

where:

E = sulfur equivalent of the KIO_3 solution in grams per milliliter,
V = milliliters of KIO_3 solution required by the sample, and
20 = 100 divided by the weight of sample used (5 g.).

NOTE 1.—In general the $ZnSO_4$ solution is preferable to the $CdCl_2$ solution because $ZnSO_4$ is more soluble in NH_4OH than is $CdCl_2$. The $CdCl_2$ solution may be used when there is doubt as to the presence of a trace of sulfide sulfur, as the yellow CdS facilitates the detection of a trace.

NOTE 2.—If the content of sulfur exceeds 0.20 or 0.25 per cent, a smaller sample should be used so that the titration with the KIO_3 solution will not exceed 25 ml.

NOTE 3.—The cooling is important, as the end point is indistinct in a warm solution. A part of the NH_4OH is lost during the distillation and the remaining NH_4OH reacts with acid, raising the temperature of the solution a few degrees without rendering the end point uncertain.

NOTE 4.—If the content of sulfur is appreciable but not approximately known in advance, the result may be low due to the loss of H_2S during a slow titration. In such a case the determination should be repeated with the titration carried out more rapidly.

NOTE 5.—Sulfites, thiosulfates, and other compounds intermediate between sulfides and sulfates are assumed to be absent. If such compounds are present, they may cause an error in the determination.

SULFUR TRIOXIDE[6]

Alternate Method

Rapid Determination by Turbidimeter

Apparatus

7. The apparatus shall consist of the following:

(*a*) *Turbidimeter.*—A standard Wagner turbidimeter, as described in the Standard Method of Test for Fineness of Portland Cement by the Turbidimeter (A.S.T.M. Designation: C 115) of the American Society for Testing Materials,[3] or a turbidimeter designed generally on the same principles. The turbidimeter should be equipped with a source of light, a water cell, a retarding filter, a sedimentation tank, a photoelectric cell, and a shield, but an elevating device is not necessary. A microammeter having a range of 0 to 50 or 100 microamperes and a source of constant emf. are required. The latter may be a 6-v. automobile starting and lighting storage battery. If the same turbidimeter is used for determining fineness and sulfur trioxide, separate sedimentation tanks shall be used for each purpose.

(*b*) *Stirring Apparatus.*—A stirring apparatus capable of maintaining a speed between 225 and 300 rpm., constant within 20 rpm. The speed may be controlled by any suitable device. An electric motor with sufficient power to maintain a constant speed and a friction drive or a system of reducing gears or pulleys make a good permanent installation. The speed, if not known for certain, may be checked by counting the rotation for 15 to 30 sec. or by means of a stroboscopic disk. A light duty laboratory electric stirrer is liable to fluctuate in speed but may be used if controlled by means of a rheostat and a stroboscope in the following manner: Fit the motor with a special shaft that extends through the upper part of the housing. On the top end of the shaft place a 1 by 5-cm. brass flywheel that carries a stroboscopic disk having a number of alternate white and black sectors (Note 1). Illuminate the disk with a neon glow lamp, the upper part of which may be painted to protect the operator's eyes from glare. The speed of stirring at which the illuminated pattern of the disk is stationary depends on the frequency of the alternating current and the number of sectors. If the current is 60-cycle, the lamp gives 7200 flashes per min. For example, a disk of 24 white and 24 black sectors gives a speed of 300 rpm. (7200 divided by 24). Adjust the rheostat until the illuminated pattern of the disk is stationary or nearly so (Note 2). If the pattern moves in the direction of rotation of the motor, the speed of the motor is 300 rpm. plus the speed of the pattern. If the pattern moves in the opposite direction, the speed of the motor is less than 300 rpm. correspondingly.

NOTE 1.—The special shaft and flywheel can be made in a machine shop or by the manufacturer of the stirrer. The operator can make his disk and paste it on the top of the flywheel.

NOTE 2.—At speeds that are multiples of the desired speed (600 rpm., 900 rpm., etc., in the example), there will be a stationary pattern, but it will be much less distinct than the pattern at the desired speed. At some speeds that are fractions of the desired speed (150 rpm., 100 rpm., etc., in the example), there will also be a stationary pattern, but it will have a greater number of sectors.

(*c*) *Stirring Rod.*—A stirring rod made of solid glass, 0.5 to 0.6 cm. in diameter and 15 to 20 cm. in length, bent at a point 4.5 to 5.5 cm. from the lower end so that the tip describes a circle 2.2 to 2.8 cm. in radius.

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[6] This method is a revision by the Working Committee on Methods of Chemical Analysis of a procedure published by Robert B. Ruddy in a paper entitled "Determination of Sulphuric Anhydride in Portland Cement by Means of the Wagner Turbidimeter," *Journal of Research*, Nat. Bureau Standards, Vol. 16, No. 6, June, 1936, p. 553 (*Research Paper RP893*). A single determination may be made in about 20 min., although it is preferable to make several determinations at a time. In case of dispute the results obtained in accordance with Section 16 of the Standard Methods of Chemical Analysis of Portland Cement (A.S.T.M. Designation: C 114 – 44) shall govern. [6]Standard Methods C 114 appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(*d*) *Measuring Scoop.*—A small scoop made from a piece of metal tubing attached to a suitable handle, and of such size as to contain 0.5 g. of the barium chloride crystals when filled. An inside diameter of 0.63 cm. and a length of 1.12 cm., giving a volume of 35 ml., may be used.

Reagent

8. *Barium Chloride Crystals.*—Use sieved crystals of $BaCl_2 \cdot 2H_2O$ that pass a No. 30 (420-micron) sieve (Note) and are retained on a No. 80 (177-micron) sieve.[7] Mix the crystals thoroughly to insure uniform distribution of crystal sizes. It is advisable to prepare a supply of crystals sufficient for a large number of determinations, because a new calibration curve must be prepared for each batch of crystals used. If the curve obtained with a new lot of crystals is closely parallel to the curve previously used, the intensity of the light may be adjusted so as to make the two curves coincide.

NOTE.—Clean sieves should be used to avoid the contamination of the crystals. Their exclusive use for the purpose is preferable.

Calibration

9. (*a*) *Standard Setting.*—The lamp of the turbidimeter shall be maintained at some standard value of intensity by adjusting the filament temperature by means of the rheostats so that the reading on the microammeter, observed with the retarding filter in place but with the sedimentation tank removed, reaches a predetermined value. This value shall be determined as follows: Using a sample of known SO_3 content, weigh an amount that gives a quantity of SO_3 corresponding to the lowest content expected to be encountered in the testing of unknown samples (Note 1). Treat the cement with HCl and proceed with the subsequent steps prescribed in Section 10. Adjust the intensity of the lamp (Note 2) so that in 60 sec. after the stirring is stopped, the reading of the microammeter is just below the upper limit of the range. Replace the retarding filter, remove the tank, and observe the reading on the microammeter, which shall constitute the standard setting.

NOTE 1: *Example.*—If the SO_3 content of the cement is 1.80 per cent and the expected minimum is 1.30 per cent, 0.3611 g. of cement shall be weighed out $\left(0.5\text{ g.} \times \frac{1.30}{1.80}\right)$. The minimum should not be less than 1.30 per cent and should be as high as practicable.

NOTE 2.—Whenever the lamp is turned on, it should be allowed to burn until the emf. is constant, as indicated by repeated readings of the microammeter taken at intervals of 5 min. The lamp shall be kept burning until the end of the work, but its intensity should be checked occasionally during the work and care should be taken not to damage the microammeter by sending it a current that greatly exceeds its range. This danger may be avoided by consistent practice of keeping either the retarding filter or a suspension in the path of the light at all times. It is important to have all contacts in the electrical circuits clean and tight. It is desirable to solder electrical connections wherever practicable. The cabinet should be closed when a reading is taken.

(*b*) *Calibration Procedure.*—Using a sample of cement of known SO_3 content, as determined by Section 16 of the Standard Methods of Chemical Analysis of Portland Cement (A.S.T.M. Designation: C 114 – 44) of the American Society for Testing Materials,[3] weigh exact amounts that give quantities of SO_3 corresponding to four or more different percentages (Note 3) in 0.5 g. of sample. Treat the specimens of cement with HCl and proceed with the subsequent steps prescribed in Section 10. Use the readings to plot a curve on graph paper (Paragraph (*c*)), each point being located by the average of at least three determinations (Note 4).

[7] Detailed requirements for these sieves are given in the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

NOTE 3.—The percentages depend on the limits in SO_3 content expected to be encountered in regular testing. Percentages of 1.30, 1.60, 1.90, 2.20, and 2.50 are suggested for a comprehensive range. The standard cement used for calibration should have an approximately average content of SO_3 (1.70 to 1.90 per cent).

NOTE 4.—When the optical system is changed in any way, such as the replacement of lamp, filter, or tank, the instrument must be recalibrated.

(*c*) *Calibration Curve.*—Plot the average readings obtained in Paragraph (*b*) against the percentage of SO_3 on semilogarithmic (one-cycle) paper, with the readings on the logarithmic axis, or plot the logarithms of the readings against the percentage of SO_3 on cross-section paper. Draw a smooth curve through the points. The curve should be nearly a straight line and slightly bent towards the origin of the axes. All curves for the same instrument should be similar and more or less parallel to each other. A chart may be prepared from the curve to enable one to read the percentage of SO_3 at a glance.

Procedure

10. (*a*) To 0.5 g. of the sample in a 150-ml. beaker, add 10 ml. of cool water and swirl the mixture until the sample is thoroughly dispersed. While continuing the swirling, add 5.0 ml. of HCl (sp. gr. 1.18). Use a glass rod to break up any lumps that do not readily dissolve. Dilute the solution immediately to 30 to 40 ml. with hot water (about 90 C.) and digest it at a temperature just short of boiling until the solution clears, which takes 5 to 10 min. (Note 1). Filter the solution into a 250-ml. beaker through a filter paper of medium porosity and wash the residue well with hot water. Dilute the filtrate and washings to 200 ml. in a graduated cylinder, adjust the temperature of the solution to any convenient temperature between 20 and 30 C. (Note 2), and return the solution to the beaker. Adjust the stirring apparatus so that the lower end of the stirring rod is 0.5 to 1.0 cm. above the bottom of the beaker and the motor is running at a speed between 225 and 300 rpm., constant within 20 rpm. (Note 3). While stirring, add 0.45 to 0.55 g. of $BaCl_2 \cdot 2H_2O$ to the solution. If the speed of the stirrer is 225 rpm., continue the stirring for 60 sec.; a period of 30 sec. is sufficient for a speed of 300 rpm. Remove the stirrer from the solution and transfer to the sedimentation tank a volume sufficient to bring the surface well above the light beam. Place the tank carefully against the guides in the instrument, remove the retarding filter, and close the cabinet. Observe the deflection of the microammeter in exactly 60 sec. after the stirring is stopped.

NOTE 1.—If the cement contains an appreciable amount of manganic oxide, there may be brown compounds of manganese that dissolve slowly in diluted HCl. They may be ignored. Take care not to lose an appreciable amount of HCl from excessive or prolonged heating.

NOTE 2.—The temperature should be the same in the calibration and determination within 2 C. The method is empirical, and it is important to give close and careful attention to all the details of the directions. Uniformity of the conditions within the specified tolerances is essential to accurate results.

NOTE 3.—It is not necessary to keep the pattern on the stroboscopic disk absolutely stationary, but it should not rotate more than 20 times in 1 min. If it begins to move, the rheostat may be slightly turned to stop the movement. The flywheel prevents sharp fluctuations. The speed of stirring may be fixed at any number from 225 to 300 rpm. and can easily be controlled within 5 rpm. with the stroboscope described in Section 7 (*b*).

(*b*) *Calculation.*—Read the percentage of SO_3 from the calibration curve or chart (Note 4).

NOTE 4.—If the reading does not fall within the limits of the calibration curve, the determination may be repeated with a smaller or larger amount of the sample as the case may require and corrected for the difference in weight.

VINSOL RESIN

Determination by the Methoxyl Method[8]

Apparatus

11. (*a*) *Semi-Micro Distillation Apparatus.*—The apparatus to be used shall be similar to the one illustrated in Fig. 1,[9] and shall consist of a reaction flask with a side arm for CO_2, an air condenser with trap, an adaptor, and two absorption tubes.

(*b*) *Pipette.*—For transferring the bromine solution to the absorption tubes, it is convenient to use a 2-ml. pipette with the stem cut off about 1 cm. below the bulb. The capacity will then be approximately 1.5 ml., and this amount can be conveniently withdrawn without suction by dipping the pipette into the bromine solution up to the mark.

(*c*) *Boiling Rod.*—To prevent bumping, a device known as a boiling rod shall be used. It shall consist of a piece of

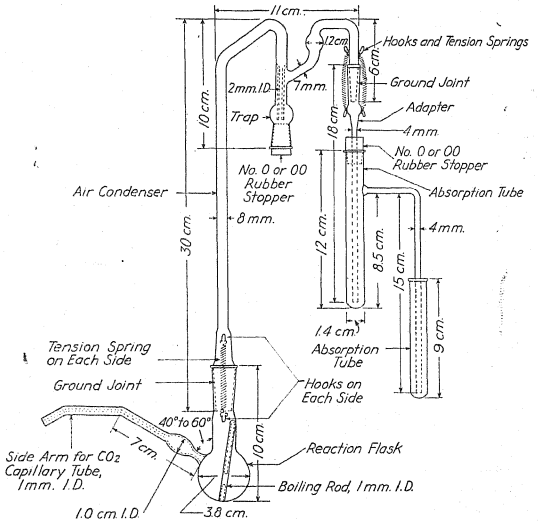


FIG. 1.—Semi-Micro Distillation Apparatus.

[8] For a discussion of the principle and historical development of the methoxyl method for determining Vinsol resin, see the paper on "The Principle of the Methoxyl Method for Determining Vinsol Resin in Portland Cement," see the compilation of "A.S.T.M. Standards on Cement," issued as a separate publication.

[9] These requirements are based on the apparatus designed by E. P. Clark, of the United States Bureau of Entomology and Plant Quarantine, for the determination of alkoxyl groups; E. P. Clark: "Studies on Gossypol. VI. The Action of Boiling Hydriodic Acid as Used in the Zeisel Method upon Gossypol and Some of its Derivatives. A Semi-Micro Zeisel Methoxyl Method," *Journal*, Am. Chemical Soc., Vol. 51, No. 5, May, 1929, pp. 1479-1483; "The Vieböck and Schwappach Method for the Determination of Methoxyl and Ethoxyl Groups," *Journal*, Assn. of Official Agricultural Chemists, Vol. 15, No. 1, February, 1932, pp. 136-140; "Semimicro Quantitative Organic Analysis," pp. 68-72, Academic Press, Inc., New York, N. Y. (1943). References to other forms of the apparatus which have been suggested for use appear in a paper on "The Principle of the Methoxyl Method for Determining Vinsol Resin in Portland Cement," see the compilation of "A.S.T.M. Standards on Cement," issued as a separate publication.

capillary glass tubing, approximately 6 cm. in length and 1 mm. in inside diameter. It shall be sealed and fire-polished at one end, and the capillary shall be closed about 1 cm. from the other end, which shall be fire-polished. When this rod is placed in the flask with the open end down, it will induce uniform boiling so long as sufficient heat is constantly applied to the liquid. A dry boiling rod shall be used for each determination. A boiling rod is not used if the side arm is a separate unit with a ground glass joint and extends close to the bottom of the reaction flask to serve the same purpose.

(*d*) *Steam Bath.*—An open-type steam bath (steam at atmospheric pressure) shall be used.

(*e*) *Oil Bath.*—An oil bath, preferably electrically heated, that can be maintained at 140 to 150 C. shall be used.

(*f*) *Burette.*—Due to the small volumes of standard $Na_2S_2O_3$ solution required for most cement samples, the use of a 5-ml. burette graduated to 0.01 or 0.02 ml. is recommended.

Reagents

12. (*a*) *Potassium Acetate in Acetic Acid.*—Dissolve 100 g. of anhydrous potassium acetate in 1 liter of glacial acetic acid.

(*b*) *Bromine in Acetic Acid.*—Add 50 ml. of liquid bromine to 200 ml. of glacial acetic acid and shake.

(*c*) *Phenol.*—Transfer some crystals of phenol to a small glass-stoppered bottle. Before using, melt the material by heating to 60 to 70 C.

(*d*) *Hydriodic Acid (57 per cent, sp. gr. 1.70).*—The blank determination on the 40-g. ignited sample, as described in Section 13(*e*), should not exceed 0.3 ml. of 0.1 *N* $Na_2S_2O_3$. If necessary, the acid may be purified by boiling with a small amount of red phosphorous and a slow stream of CO_2 for 20 to 30 min., followed by distillation at 126 to 127 C. in all-glass apparatus. A slow stream of CO_2 should be maintained through the receiver during the distillation. During the distillation, the poisonous gas phosphine, PH_3, may be formed under certain conditions, and this may unite with molecular iodine to form the explosive PI_3, similar in properties to NI_3. The PI_3 may explode on contact with air. It is therefore advisable to keep the current of CO_2 going after the distillation has been finished, until the apparatus has cooled; this prevents air from being sucked into the apparatus. As a safety precaution, the distillation should be conducted in a hood or behind a wire-glass shield.

(*e*) *Carbon Dioxide.*—A Kipp generator with the use of marble and HCl (1:1) is convenient, or a cylinder of the gas equipped with a suitable needle valve may be used. In either case, it is desirable to pass the gas through a wash bottle containing saturated $NaHCO_3$ solution. Gaseous nitrogen may be substituted for CO_2.

(*f*) *Sodium Acetate Solution.*—Dissolve 250 g. of anhydrous sodium acetate in 1 liter of water.

(*g*) *Formic Acid (90 per cent).*

(*h*) *Starch Solution.*—To 500 ml. of boiling water, add a cold suspension of 5 g. of soluble starch in 25 ml. of water. Cool, add a cool solution of 5 g. of NaOH in 50 ml. of water, add 15 g. of KI, and mix thoroughly.

(*i*) *Standard Sodium Thiosulfate (approximately 0.1 N).*—Dissolve 25 g. of $Na_2S_2O_3 \cdot 5H_2O$ in 200 ml. of water and dilute to 1 liter. Use freshly boiled and cooled water. It is preferable to allow the solution to stand for a few days before standardization. Standardize the solution against an approximately 0.1 *N* solution of $KMnO_4$ that has been standardized against sodium oxalate oxidimetric standard furnished by the Na-

tional Bureau of Standards (standard sample No. 40). The standard 0.18 *N* potassium permanganate described in Section 31 of the Standard Methods of Chemical Analysis of Portland Cement (A.S.T.M. Designation: C 114 – 44)[3] may be used if desired. Proceed with the standardization as follows: In each of two glass-stoppered Erlenmeyer flasks put 2 g. of KI and 100 ml. of freshly boiled and cooled water. Shake to dissolve the KI and add 2 ml. of HCl (sp. gr. 1.18). To one flask add slowly from a burette either 20.0 ml. of 0.1 *N* $KMnO_4$ or 10.0 ml. of 0.18 *N* $KMnO_4$, while swirling the flask gently. To the other flask add an equal volume of water. Stopper the flasks and let them stand in the dark for 10 min. Titrate the iodine that has been set free with the $Na_2S_2O_3$ until the solution is of a faint straw color. Add 2 ml. of starch solution and continue the titration until the blue color has just been destroyed. Subtract the volume of $Na_2S_2O_3$ required in the blank determination from that required in the other titration, and calculate the normality of the $Na_2S_2O_3$, based on the normality of the $KMnO_4$. Calculate the Vinsol resin value of the $Na_2S_2O_3$ solution as follows:

$$E = N \times 0.2716$$

where:

E = value of 1 ml. of the $Na_2S_2O_3$ solution in terms of percentage of Vinsol resin in a 40-g. sample of cement,

N = normality of the solution, and

0.2716 = Vinsol resin value of an exactly 1 *N* $Na_2S_2O_3$ solution.[8]

Procedure

13. (*a*) Extract the chloroform-soluble organic substances from 40 g. of cement as described in Section 27 of the Standard Methods of Chemical Analysis of Portland Cement (A.S.T.M. Designation: C 114 – 44).[3] After weighing the chloroform extract (Note 1), redissolve it in a little chloroform. It may be necessary to warm the chloroform slightly to effect complete solution rapidly. If trouble is encountered in complete solution, the addition of a little acetone may assist the solution. Quantitatively transfer the solution to the methoxyl reaction flask, using a minimum of chloroform for the transfer. Evaporate the solvent on the steam bath, taking care not to prolong the heating after the residue appears dry. A stream of dry air directed down into the flask or through the capillary will hasten the evaporation. After the residue appears dry, heat the flask and contents in an oven at 100 to 105 C. for 15 min.

NOTE 1.—The procedure described in Paragraph (*a*) assumes that values for both total chloroform-soluble organic substances and for Vinsol resin are desired on the sample. If only the value for Vinsol resin is desired, the chloroform solution obtained in Section 27 (*d*) of Standard Methods C 114 may be filtered directly into the methoxyl reaction flask instead of into a weighed beaker or platinum dish. In this case, proceed with the evaporation of the solution in the flask as described in Paragraph (*a*) of this section.

(*b*) Add approximately 6 ml. of the solution of potassium acetate in acetic acid to the first absorption tube and 4 ml. to the second tube. By means of the shortened 2-ml. pipette, add approximately 1 ml. of the solution of bromine in acetic acid to the first absorption tube and 0.5 ml. to the second. Stopper the tubes temporarily to prevent the bromine fumes from escaping into the air. Invert the condenser and add a pinch (about twice the size of a pin head) of red phosphorus to the trap; then add enough water so that when the rubber stopper is inserted and the condenser is placed upright, the capillary tube will dip just below the surface of the liquid.

Add 2.5 ml. of melted phenol by means of a warm pipette and 5 ml. of HI (57 per cent, sp. gr. 1.70) (Note 2), and then a dry boiling rod, to the flask containing the sample. Moisten the ground-glass joint with a drop of HI, and connect the flask and condenser. Connect the adapter to the other end of the condenser, moistening the ground-glass joint with a drop of glacial acetic acid. Place tension springs or rubber bands around the hooks at the ground-glass joints. Connect the two absorption tubes, using a one-hole rubber stopper at the top of the first tube and a piece of moistened absorbent cotton around the glass tubing at the top of the second tube to minimize the escape of noxious bromine fumes into the air.

NOTE 2.—As the HI sometimes has a rather high blank value, it is advisable to measure the 5 ml. of acid as accurately as possible from a pipette.

(*c*) Connect the apparatus to a source of CO_2 and place in an oil bath at 140 to 150 C. Pass a slow current of CO_2 through the apparatus at a rate of about two bubbles per second, and maintain the oil bath at a temperature of 140 to 150 C. At the end of about 15 min., increase the current of CO_2 to about three bubbles per second, and continue for an additional 15 min., or a total of 30 min.

(*d*) Add 5 ml. of the sodium acetate solution to a 250-ml. Erlenmeyer flask. Transfer the contents of both absorption tubes quantitatively to the flask, using water. Dilute to 100 to 125 ml. with water, and add formic acid (90 per cent) drop by drop, while swirling, until the brown color of bromine is discharged; then add 10 to 12 drops in excess. Allow the solution to stand 1 to 2 min., and remove any bromine vapor that may remain in the flask by blowing a gentle stream of CO_2 over the liquid. Add 2 g. of KI, and swirl the flask to dissolve the KI. Add 5 ml. of H_2SO_4 (6:94) and titrate immediately with the standard $Na_2S_2O_3$ solution to a light straw color. Add 2 ml. of starch solution and continue the titration to the disappearance of the blue color.

(*e*) *Blank.*—Make a blank determination with 40 g. of ignited cement (Note 3), following the procedure described in Section 27(*e*) of Standard Methods C 114 and then using the same amounts of reagents and procedure as prescribed in Paragraphs (*a*) to (*d*) of this section.

NOTE 3.—It is not necessary to make a blank determination on ignited samples of each cement when determinations are made on a series of cement samples. However, a blank determination should be made on each lot of chloroform or HI.

(*f*) *Calculation.*—Calculate the percentage of Vinsol resin as follows, and report the results to the nearest 0.001 per cent:

$$\text{Vinsol resin, per cent} = E(V_1 - V_2)$$

where:

E = value of 1 ml. of the $Na_2S_2O_3$ solution in terms of percentage of Vinsol resin in a 40-g. sample of cement (see Section 12(*i*)),

V_1 = milliliters of $Na_2S_2O_3$ solution required by the sample, and

V_2 = milliliters of $Na_2S_2O_3$ solution required by the blank.

Tentative Method of Test for
AIR CONTENT OF PORTLAND-CEMENT MORTAR[1]

A.S.T.M. Designation: C 185 – 44 T

ISSUED, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. This method of test is intended for determining the air content of portland-cement mortar under the conditions hereinafter specified.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Measure.*—A cylindrical measure having an inside diameter of $3 \pm \frac{1}{16}$ in. and a depth (approximately $4\frac{5}{16}$ in.) adjusted by standardization with water to contain 500 ± 1 ml. at 21.1 C. (70 F.) (Note 1). A satisfactory mold may be machined from seamless steel tubing or nominal 3-in. wrought-iron pipe, to which a steel disk about $\frac{1}{8}$ in. in thickness is welded to form the bottom.

NOTE 1.—The 500-ml. measure can be calibrated readily by filling with distilled water at 21.1 C. (70 F.) to a point where the meniscus extends appreciably above the top of the measure, placing a clean piece of plate glass on the top of the measure, and allowing the excess water to be squeezed out. The absence of air bubbles as seen through the glass insures that the measure is completely full. Care shall be taken that the excess water is wiped from the sides of the container before weighing.

Another method of calibration is to cover the measure, which has been set on counter-balanced scales, with the clean glass plate. The measure shall be level during the calibration. The plate shall be set over the top of the measure so that only a narrow slit remains uncovered. Distilled water at 21.1 C. (70 F.) shall then be poured through the opening between cover and measure until the water is in contact with the glass plate at all points. The water level in the slit shall then be adjusted so that there is neither a positive nor negative meniscus with the edge of the cup. This is easily observed by noting reflection of light, with the eye at the level of the cup.

In both methods, the water content of the measure, in grams, divided by 0.998 gives the capacity in milliliters.

(*b*) *Tamping Rod.*—A bullet-pointed rod $\frac{1}{4}$ in. in diameter and 10 in. in length.

(*c*) *Straightedge.*—A steel straightedge not over $\frac{1}{4}$ in. in thickness.

(*d*) *Spatula.*—A 6-in. metal spatula with straight edges.

(*e*) *Scales.*—Scales having a capacity sufficient to weigh the mortar and the container (approximately 2 kg.). The

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-1 on Cement.

[2] Accepted by the Society at annual meeting, June, 1944.

permissible variation at 2 kg. shall be plus or minus 1 g.

(*f*) *Mixing Bowl.*—A mixing bowl of 4-qt. capacity. A circular metal or enameled bowl with a flat bottom is preferred.

(*g*) *Sieves.*—Square-hole, woven wire cloth, No. 20 (840-micron) and No. 30 (590-micron) sieves conforming to the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11).[3]

(*h*) *Glass Graduates.*—Glass graduates of 250-ml. capacities, for measuring the mixing water, made to deliver the indicated volume at 20 C. (68 F.). The permissible variation shall be plus or minus 2 ml. The main graduation lines shall be circles and shall be numbered. The least graduations shall extend at least one seventh of the way around and intermediate graduations shall extend at least one fifth of the way around.

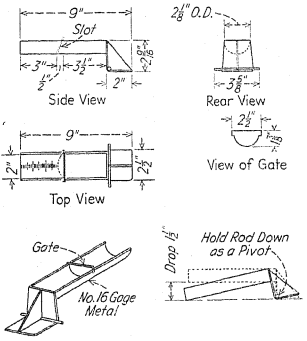


FIG. 1.—Burmister Mortar Flow Trough.

(*i*) *Burmister Mortar Flow Trough.*—A Burmister mortar flow trough conforming to the dimensions shown in Fig. 1 and made of sheet metal. The trough shall be mounted or operated on a rigid base or table (Note 2).

NOTE 2.—The mortar flow trough may be operated advantageously by means of a cam operating against the heel of the base plate.

Temperature

3. The temperature of the room and dry materials shall be maintained between 20 and 27.5 C. (68 and 81.5 F.). The temperature of the mixing water shall not vary from 21 C. (70 F.) by more than plus or minus 1.7 C. (3 F.).

Standard Sand

4. The sand used for making the standard mortar (Section 5 (*a*)) shall be natural silica sand from Ottawa, Ill., graded to pass a No. 20 (840-micron) sieve and retained on a No. 30 (590-micron) sieve. This sand shall be considered standard when not more than 15 g. are retained on the No. 20 sieve and not more than 5 g. pass the No. 30 sieve after 5 min. of continuous sieving of a 100-g. sample in the manner specified for sieving cement in the Standard Method of Test for Fineness of Hydraulic Cement by the No. 200 Sieve (A.S.T.M. Designation: C 184).[3]

Procedure

5. (*a*) *Batch.*—The proportions of the standard mortar shall be 300 g. of cement to 1200 g. of standard sand and sufficient water, in multiples of 4 ml., to give a flow between 2.4 and 2.9 in. (Note 1) on the mortar flow trough when determined in accordance with Paragraph (*c*).

NOTE 1.—It is indicated by test that the air content of the mortar is not affected appreciably by a variation in flow within the 0.5-in. range specified.

(*b*) *Mixing of Mortar.*—The mixing shall be done in a bowl (Section 2 (*f*)) by vigorous and continuous stirring, squeezing, and kneading with one hand, which

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

shall be protected by a snug-fitting rubber glove. The materials for a batch shall be introduced in the following manner:

(*1*) Place the water in the bowl, which has just been wiped with a damp cloth or damp sponge,
(*2*) Add the cement to the water and mix for 30 sec.,
(*3*) Add approximately one half of the standard sand and mix for 30 sec.,
(*4*) Add the remainder of the sand and mix for 1 min.,
(5) Let the mortar stand for 1½ min. (covered with a damp cloth), and
(6) Mix for 1 min.

(*c*) *Flow Determination.*—The mortar flow trough shall be dry at the beginning of the flow determination. The gate shall be put in place and the trough supported in the horizontal position. The trough shall be filled between the gate and the closed end with the mortar in two layers, puddling each layer 15 times with the gloved forefinger of one hand. The remaining mortar of the batch in the bowl shall be covered with a damp cloth. Care shall be taken in placing the mortar under the sloping gate and at the back end of the trough. Immediately after completion of puddling the last layer and before smoothing off the surface, the closed end of the trough shall be tapped lightly twice on each side with the handle of the spatula to prevent the mortar from sliding and breaking away from the closed end during the flow determination. The flat side of the spatula blade shall be placed across the trough just behind the gate, with the length of the blade at right angles to the length of the trough. The mortar shall then be smoothed off by drawing the spatula blade, with its leading edge slightly raised, to the closed end of the trough in a single stroke. The mortar shall then be cut off to a level surface flush with the top of the trough by drawing the straight edge of the spatula with a sawing motion over the trough from the gate to the back end. Care shall be taken that the surface of the trough from the gate to the open end is clean and dry. If seepage under the gate has occurred, it shall be removed with a dry absorbent cloth. The gate shall be removed by lifting one side in a hinge motion, using the other side as a pivot, and the free end of the trough shall be allowed to drop under its own weight. The free end shall again be raised until the trough is horizontal, and again allowed to drop. Ten drops shall be made at the rate of 1 drop per second and the flow recorded to the nearest 0.1 in. The operation of raising and dropping the free end of the flow trough shall be started immediately after removing the gate and not more than 45 sec. after completion of mixing. Each determination of flow shall be made on a fresh portion of material from the same or a new batch. When the flow is not within the required range (paragraph (*d*)), a new batch shall be made with an adjusted water content.

(*d*) *Weight per 500 ml. of mortar.*—When the quantity of mixing water has been found that produces a flow between 2.4 and 2.9 in., the weight per 500 ml. of mortar shall be determined immediately, using the mortar remaining in the mixing bowl after the flow has been determined. The portion of the mortar used in the flow determination shall not be used in the determination of the weight per 500 ml. The mortar shall be placed in the 500-ml. measure in three equal layers, rodding each layer 15 times with the tamping rod and taking care to penetrate only the surface of the layer previously placed. Each layer shall be spaded thoroughly with the spatula around the inner surface of the measure to remove all rod holes and to consolidate the mortar adhering to the inner surface of the measure. Care shall be taken that no rod holes are left in the mortar and

that no additional holes are introduced by the spading operation. The excess mortar shall be struck off with a steel straightedge flush with the top of the measure, making two passes over the entire surface, the second pass being made at right angles to the first. Care shall be taken in the striking-off operation that no loose sand grains cause the straightedge to ride above the top surface of the measure. The entire operation of filling and striking off the measure shall be accomplished within 1½ min. All mortar and water adhering to the outside of the measure shall be wiped off and the measure and contents weighed (Note 2). The weight of the mortar in grams, after subtracting the weight of container, shall be recorded.

NOTE 2.—This operation may be facilitated by placing the measure on a steady flat-surfaced support of lesser diameter than the measure while filling and wiping.

Calculation

6. The air content of the mortar shall be calculated from the following formula, which is based on the batch proportions given in Section 5 (*a*), taking the specific gravity of cement as 3.15 and of standard Ottawa sand as 2.65:[4]

Air content, per cent by volume =

$$100 - 2W\,\frac{(182.7 + P)}{(5000 + 10P)}$$

where:

W = weight of 500 ml. of mortar in grams, and

P = percentage mixing water based on weight of cement used.

Reproducibility

7. Duplicate determinations of the air content shall be made by this method on separate batches and the average value obtained shall be reported. Values for percentage of air content obtained in the test should not differ from the average by more than plus or minus 1.0.

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[4] This formula may be derived as follows:

$$\text{Air content, per cent by volume} = 100\left(1 - \frac{W_a}{W_c}\right) \quad . . (1)$$

where:

W_a = actual weight per unit of volume as determined by A.S.T.M. Method C 185,

$= \frac{W}{500}$ g. per ml., where W is the weight in grams of the specified 500 ml. of mortar (Section 5(*d*)),

W_c = theoretical weight per unit of volume, calculated on an air-free basis as follows, and using the values for specific gravities and quantities of materials as given in Sections 5(*a*) and 6,

$$= \frac{300 + 1200 + 300 \times P \times 0.01}{\frac{300}{3.15} + \frac{1200}{2.65} + \frac{300 \times P \times 0.01}{1}}$$

$$= \frac{5 + 0.01P}{1.827 + 0.01P}, \text{ and}$$

P = percentage of mixing water, based on weight of cement.

Substituting for W_a and W_c in Eq. 1, we have:

Air content, per cent by volume

$$= 100\left(1 - \frac{W}{500} \times \frac{1.827 + 0.01P}{5 + 0.01P}\right)$$

$$= 100 - \frac{W}{5} \times \frac{(1.827 + 0.01P)}{(5 + 0.01P)}$$

$$= 100 - 2W\,\frac{(182.7 + P)}{(5000 + 10P)}$$

Tentative Method of Test for

HEAT OF HYDRATION OF PORTLAND CEMENT[1]

A.S.T.M. Designation: C 186 – 44 T

ISSUED, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. This method of test is intended for determining the heat of hydration of a cement by measuring the heat of solution of the dry cement and the heat of solution of a separate portion of the cement that has been partially hydrated for 7 or 28 days, the difference between these values being the heat of hydration for the respective period of hydrating.

Apparatus

2. (*a*) *Calorimetric Apparatus*:

(*1*) *Calorimeter.*—The calorimeter (Fig. 1) shall consist of a 1-pt., wide-mouth vacuum jar, with cork stopper, held in a suitably insulated container (see item (*2*)) to keep the vacuum jar in position and to protect the jar from undue temperature fluctuations. The vacuum jar shall not cool more than 0.001 C. per min. per degree of difference from room temperature when filled with warm water, stoppered, and allowed to stand unstirred for 30 min. The vacuum jar shall be coated on the interior with a material resistant to hydrofluoric acid, such as a baked phenolic resin, a baked vinyl chloride acetate resin, or beeswax. The acid-resistant coating shall be intact and free of cracks at all times; it shall be examined frequently and renewed whenever necessary.

(*2*) *Insulated Container.*—The container shall have an insulating layer of cotton or similar material, which shall be at least 1 in. in thickness and shall encase the sides and bottom of the vacuum jar, but shall be so arranged as to permit the easy removal of the jar.

(*3*) *Differential Thermometer.*—The adjustable differential thermometer shall be of the Beckmann type, graduated at least to 0.01 C., and shall have a range of approximately 6 C. The thermometer shall be so adjusted that the upper limit of the scale approximates room temperature. The portion of the thermometer that will rest inside the calorimeter shall be protected with a coating resistant to hydrofluoric acid (see item (*1*)). The thermometer shall be equipped with a suitable reading lens.

(*4*) *Funnel.*—The funnel through which the sample is introduced into the

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-1 on Cement.

[2] Adopted by the Society at annual meeting, June, 1944

calorimeter shall be of glass or plastic and shall have a stem approximately 3 in. in length and with an inside diameter of not less than $\frac{1}{4}$ in.

(5) *Stirrer.*—The stirrer shall be made of glass with a coating resistant to hydrofluoric acid, or of an inert plastic, and shall be long enough to extend nearly to the bottom of the calorimeter. The motor shall be of the constant-speed type, at least $\frac{1}{20}$ hp., and shall be equipped with a geared speed reducer so that some one speed, in the range of 350 to 700 rpm., can be maintained constant (Note).

NOTE.—The function of the stirrer is twofold: to maintain uniform temperature throughout the liquid and to supply sufficient agitation to keep the solid reactant in suspension in the acid mixture. In this calorimeter the latter function controls the design of the stirrer, which may be varied by the operator to give satisfactory performance. Since a stirrer capable of keeping the solid in suspension generates considerable heat in the calorimeter, it is important that the stirrer speed, and hence the rate of heat generation, be maintained constant. A synchronous motor and a geared speed reducer are therefore almost mandatory.

(*b*) *Mixer.*—A mechanical mixer, such as a milk-shake stirrer, capable of intimately mixing the cement and water to a uniform paste.

(*c*) *Storage.*—Storage space with temperature controlled at 21 ± 1.7 C.

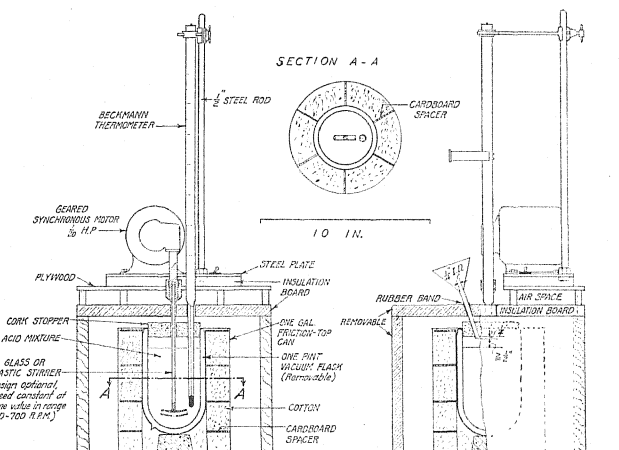


FIG. 1.—Calorimeter.

(*d*) *Mortar.*—A mortar, approximately 8 in. in diameter, and pestle for grinding the partially hydrated samples.

(*e*) *Glass Vials.*—Glass vials, 4 by $\frac{3}{4}$ in., shell type, with stoppers.

(*f*) *Oven.*—A drying oven maintained at 100 to 110 C.

(*g*) *Weighing Bottles.*

(*h*) *Stop Watch or Clock Timer.*

(*i*) *Sieves.*—No. 100 (149-micron) and No. 30 (590-micron) sieves conforming to

the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11).[3]

(*j*) *Crucibles.*—Platinum crucibles of 30-ml. capacity, with covers, for loss on ignition determinations.

(*k*) *Muffle.*—A muffle furnace or suitable burners capable of maintaining a temperature of 900 to 950 C.

(*l*) *Analytical Balance.*—An analytical balance and analytical weights, conforming to the requirements prescribed in the Standard Methods of Chemical Analysis of Portland Cement (A.S.T.M. Designation: C 114),[3] for weighing out calorimetric samples and for loss on ignition weighings.

(*m*) *Balance.*—A glass enclosed balance, capacity 1 kg., capable of weighing the acid charge to the nearest 0.05 g.

Reagents

3. (*a*) *Zinc Oxide.*—Reagent grade ZnO, for use as the standard in the heat capacity determinations, shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society. The ZnO shall be heated at 900 to 950 C. for 1 hr., then cooled in a desiccator, ground to pass a No. 100 (149-micron) sieve, and stored. Immediately prior to the heat capacity determinations, 7 g. of the ZnO so prepared shall be heated for not more than 5 min. at 900 to 950 C., cooled to room temperature in a desiccator, and weighed accurately for introduction into the calorimeter (Note).

NOTE.—The rate of solution of the zinc oxide varies with the preliminary treatment. The procedure described results in a product which dissolves at about the same rate as the dry cement.

(*b*) *Hydrofluoric Acid (48 per cent).*

(*c*) *Nitric Acid (2.00 N).*—The 2.00 *N* HNO_3, for use in the calorimeter, shall be prepared and standardized in large quantities. Optionally, the diluted HNO_3 may be made up with 127 ml. of HNO_3 (sp.gr. 1.42) per liter of solution, provided that heat capacity determinations are made with each batch of diluted HNO_3 so prepared.

(*d*) *Wax.*—Paraffin wax, or other suitable wax, for sealing vials.

Procedure for Determining Heat Capacity of Apparatus

4. (*a*) Determine the heat capacity of the system (that is, the number of calories required to raise the temperature of the calorimeter and contents 1 C.) by measuring the corrected temperature rise obtained by dissolving 7 g. of ignited ZnO in the specified acid mixture (Paragraphs (*b*) to (*g*)).

(*b*) Transfer approximately 400 g. of the 2.00 *N* HNO_3, which has been cooled to the temperature indicated by the lower range of the Beckmann thermometer (ordinarily about 4 to 5 C. below room temperature), into the vacuum jar, add 8.0 ml. of HF (48 per cent), weigh, and add sufficient additional 2.00 *N* HNO_3 to bring the total weight of the solution to 425.0 g. Then assemble the calorimeter and start the stirring motor. Take care that the stirrer blade does not touch the thermometer, the sides of the jar, or the cork stopper. The lower end of the funnel stem shall extend approximately $\frac{1}{4}$ in. below the lower surface of the stopper and at least $\frac{1}{2}$ in. above the level of the liquid. After an initial stirring period of at least 15 min. to allow the temperature of the system to become uniform, observe and record the temperature to the nearest 0.001 C. and record the time. After a preliminary rating period of at least 5 min., again observe and record the temperature to the nearest 0.001 C., record the time, and immediately introduce the prepared ZnO

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

through the funnel at a uniform rate (Note 1). Complete the introduction of the ZnO in 1 to 2 min. Brush any ZnO clinging to the funnel stem into the acid mixture by means of a small camel's-hair brush.

NOTE 1.—It is assumed throughout that the temperature of a sample is identical with that of the room when the sample is introduced into the calorimeter.

(*c*) Make temperature readings every minute for the first 5 min. and every 5 min. thereafter until a constant rate of temperature change is attained. When the temperature changes occurring during three successive 5-min. intervals do not differ by more than 0.001 C. from their average expressed to the nearest 0.001 C., the rate of temperature change shall be considered constant. The period from the time the introduction of the ZnO is started to the time the rate of temperature change becomes constant is termed the solution period and should last not longer than 30 min. Discard determinations in which the solution period is more than 30 min.

(*d*) The period immediately following the solution period is known as the final rating period. To correct for heat of stirring and the thermal leakage into or out of the calorimeter, calculate the corrected temperature rise as follows:

$$R = R_o - V_r T_x + K(\theta_x T_x - \theta_i T_x)$$

where:

R = corrected temperature rise in degrees Centigrade,
R_o = observed temperature rise in degrees Centigrade,
V_r = rate of temperature change during the final rating period in degrees Centigrade per 5-min. interval,
T_x = number of 5-min. intervals in the solution period,
K = leakage constant in degrees Centigrade per 5 min. per degree temperature difference, and
θ_x and θ_r = average calorimeter temperatures during the solution and final rating periods, respectively.

Calculate the leakage constant, K, as follows:

$$K = \frac{V_a - V_r}{\theta_r - \theta_a}$$

where:

V_a and θ_a = rate of temperature change per 5 min. and the average temperature, respectively, during the preliminary rating period.

Calculate the product $\theta_x T_x$ as follows:

$$\theta_x T_x = 0.1\,\theta_i + 0.2(\theta_1 + \theta_2 + \theta_3 + \theta_4 + 3\,\theta_5) + \theta_{10} + \cdots + 0.5\,\theta_n$$

where:

θ = temperature of the calorimeter and the subscripts indicate the time at which the temperature was recorded, and
θ_i and θ_n = initial and final temperatures, respectively, of the solution period.

(*e*) Calculate the heat capacity of the calorimeter and contents as follows (Note 2):

$$C = \frac{W(256.1 + 0.1(30 - t) + 0.12(T - t))}{R}$$

where:

C = heat capacity in calories per degree Centigrade,
W = weight of ZnO in grams,
t = final temperature in degrees Centigrade of the calorimeter,
T = temperature in degrees Centigrade of the ZnO (room temperature) when introduced into the calorimeter, and
R = corrected temperature rise in degrees Centigrade.

NOTE 2.—The heat of solution of ZnO is 256.1 cal. per g. at 30 C. This value increases 0.1 cal. for each degree decrease in temperature below 30 C. The heat capacity of ZnO is 0.12 cal. per g. The heat required to bring the ZnO to the final temperature of the calorimeter must be included in the effective heat of solution.

(*f*) If more than a trace of ZnO is found adhering to the tip of the funnel or to the stopper when the calorimeter is opened, the test shall be rejected.

(*g*) Redetermine the heat capacity at the following times:

(*1*) When the Beckmann thermometer is reset,

(*2*) When a new coating is applied to thermometer, stirrer, or flask,

(*3*) When a new thermometer, stirrer, or flask is put in service,

(*4*) When a new batch of acid is used, and

(*5*) At other times when, according to the judgment of the operator, the need is indicated.

Procedure for Determining Heat of Hydration

5. (*a*) *Preparation of Cement Paste.*—Immediately before mixing the cement paste, the temperature of the cement and the water shall be brought to 21 ± 1.7 C. Mix 150 g. of cement and 60 ml. of distilled water by means of a spatula, and then vigorously stir the mixture with a mechanical stirrer for 5 min. Place approximately equal representative portions of the paste in four or more glass vials, filling the vials to within about 1 in. of the top. Immediately after filling the vials, close them with tight-fitting stoppers inserted far enough into the vials to leave a space of at least ¼ in. between the top of the stopper and the top end of the vial. Fill this space with molten wax to further seal the specimens. Store the vials in an upright position at 21 ± 1.7 C. until the time of test.

(*b*) *Preparation of Partially Hydrated Sample for Heat of Solution Test.*—At the specified age of test, remove a vial of the partially hydrated sample from storage and, during a 15-min. initial stirring period of the calorimeter, break the glass away from the sample and rapidly crush the entire sample with a mortar and pestle so that all the material will pass through a No. 30 (590-micron) sieve; then quickly place the sample in a well-stoppered weighing bottle. Take care, particularly with the 7-day partially hydrated sample, to expose the sample to the air as little as possible, and thus minimize the action of CO_2 or the loss of moisture from the sample.

(*c*) *Calorimetric Procedure, Dry Cement.*—Determine the heat of solution of the dry cement sample according to the procedure described for the heat capacity determination (Section 4), but use a 3-g. sample (weighed to the nearest 0.001 g.) of the dry cement instead of the prepared ZnO (see Note 1 under Section 4(*b*)). (As in the case of a sample for chemical analysis, great care shall be exercised in securing a uniform and representative sample.) Calculate and report the results on the ignited weight basis (Paragraph (*e*)). Make the heat of solution test on the dry cement just prior to the test on the corresponding 7-day partially hydrated sample.

(*d*) *Calorimetric Procedure, Partially Hydrated Sample.*—For the heat of solution of the partially hydrated sample, follow the same procedure as for the dry cement described in Paragraph (*c*), but use a 4.18 ± 0.05 g. calorimetric sample of the partially hydrated cement, weighed to the nearest 0.001 g. (see Note 1 under Section 4(*b*)). Calculate the results on the ignited basis.

(*e*) *Loss on Ignition.*—At the same time the calorimetric sample is being weighed out, weigh a sample of similar amount into a platinum crucible for determination of loss on ignition. Ignite the dry cement at 900 to 950 C. for at

least 1½ hr. Immediately place the crucible containing the sample in a desiccator and allow to cool to room temperature; then quickly weigh the sample. When determining the loss on ignition of the hydrated cement, first dry the weighed sample in an oven at 100 to 110 C. for 1 hr.; then place the sample in a muffle furnace at 900 to 950 C. overnight, or over a suitable burner for not less than 5 hr. Reduce the weight of the cement sample that was introduced into the calorimeter to the ignited weight basis for use in the final calculations as follows:

$$W_i = \frac{A}{B} \times W$$

where:

W_i = weight of calorimetric sample in grams, on ignited basis,
A = weight of ignited sample,
B = weight of sample before ignition, and
W = weight of calorimetric sample.

Calculations

6. (*a*) *Heat of Solution of Dry Cement.*—Determine the heat of stirring and the thermal leakage correction and apply those corrections to the observed temperature rise, exactly as described in the heat capacity determination (Section 4). Also, correct the heat of solution value if the final calorimeter temperature of the heat of solution test is different from the temperature of the calorimetric sample when introduced. Thus, for the dry cement, which has a specific heat of approximately 0.2 cal., if the final calorimeter temperature *exceeds* the temperature of the cement sample at the time it was introduced, *add* a correction of 0.2 cal. per g. per degree Centigrade difference in those temperatures when calculating the heat of solution. Calculate the heat of solution of the dry cement as follows:

$$H_1 = \frac{RC}{W_i} - 0.2(T - t_d)$$

where:

H_1 = heat of solution of dry cement in calories per gram,
R = corrected temperature rise in degrees Centigrade,
C = heat capacity in calories per degree Centigrade,
W_i = weight of sample in grams on ignited basis,
T = room temperature in degrees Centigrade, when sample is introduced, and
t_d = final calorimeter temperature in degrees Centigrade at end of determination on dry cement.

(*b*) *Heat of Solution of Partially Hydrated Sample.*—Calculate the heat of solution of the partially hydrated sample in the same way as for the dry cement (Paragraph (*a*)), except make additional corrections, as follows:

(*1*) Since an increase of 1 C. in the temperature at which the heat of solution test occurs causes a decrease of approximately 0.3 cal. per g. in the heat of solution, if the temperature of the heat of solution test of the partially hydrated sample *exceeds* the temperature of the dry cement determination, a correction of 0.3 cal. per g. per degree Centigrade difference in temperature shall be *added* to the heat of solution value obtained for the partially hydrated sample.

(*2*) Also, correct the heat of solution value if the final calorimeter temperature of the solution test is different from the temperature of the calorimetric sample when introduced. Thus, for the partially hydrated sample, which has a specific heat of approximately 0.4 cal. per gram of ignited cement, if the final calorimeter temperature *exceeds* the temperature of the sample at the time it was introduced, *add* a correction of 0.4 cal. per gram of the ignited cement per degree

Centigrade difference in those temperatures when calculating the heat of solution.

Calculate the heat of solution of the partially hydrated sample as follows:

$$H_2 = \frac{RC}{W_s} - 0.4(T - t_h) - 0.3(t_d - t_h)$$

where:

H_2 = heat of solution of partially hydrated sample in calories per gram,

R, C, W_s, T, and t_d = the same as in Paragraph (*a*), and

t_h = final calorimeter temperature in degrees Centigrade at end of determination on partially hydrated sample.

(*c*) *Heat of Hydration.*—Calculate the heat of hydration of the cement to the nearest calorie, as follows:

$$H_h = H_1 - H_2$$

where:

H_h = heat of hydration, in calories per gram of ignited cement,

H_1 = heat of solution of the dry cement (Paragraph (*a*)), and

H_2 = heat of solution of the partially hydrated sample (Paragraph (*b*)).

(*d*) *Final Temperature Correction.*—A final calorimeter temperature of 25 C. shall be considered as the basis to which the heat of hydration shall be referred, and the effects of variation in that temperature should be kept in mind when considering test results. An *increase* in the final temperature *raises* the heat of hydration approximately 0.1 cal. per deg. Cent. per gram of ignited cement. For example, if the final temperature is 27 C., 0.2 cal. should be subtracted from the observed heat of hydration in order to refer the results to 25 C. In borderline cases, proper correction shall be made for the effects of final calorimeter temperature.

Tentative Methods of

PHYSICAL TESTING OF QUICKLIME AND HYDRATED LIME[1]

A.S.T.M. Designation: C 110 – 38 T

ISSUED, 1934; REVISED, 1938.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These methods cover physical tests for quicklime and hydrated lime.

RESIDUE

Apparatus

2. The sieves used shall conform to the requirements of the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials.[3]

Residue of Quicklime

3. A representative 2½-kg. (5-lb.) sample of the quicklime shall be selected. Lime selected for this test shall be broken so as to entirely pass a 1-in. ring and be retained on a ¼-in. sieve. Pulverized lime shall be tested as received. The sample shall be placed in a box of wood or of some material of similarly low thermal conductivity, and slaked by an experienced operator with sufficient water at 70 to 80 F. (21 to 27 C.) to produce the maximum quantity of lime putty, care being taken to avoid "burning" or "drowning" the lime. It shall be allowed to stand for 1 hr. and then washed through a No. 20 (840-micron) sieve by a stream of water having a moderate pressure. No material shall be rubbed through the sieve. The washing shall be continued until the residue on the screen appears to consist entirely of coarse particles, but in no case shall the washing be continued for more than 30 min. The residue shall be dried to constant weight at a temperature of 212 to 225 F. (100 to 107 C.) and the percentage residue calculated, based on the original weight of the sample.

Residue of Hydrated Lime

4. A 100-g. sample of the hydrated lime as received shall be selected and placed on a No. 30 (590-micron) sieve, which shall be nested above a No. 200 (74-micron) sieve. The material shall be washed through the sieves by means of a stream of water from a faucet. A piece of rubber tubing attached to the water faucet shall be used for the washing. The velocity of the water may be

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee C-7 on Lime.

[2] Revision accepted by the Society at annual meeting, June, 1938. Editorially revised in 1939 and 1942.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

increased by pinching the tubing, but shall not be sufficient to cause any splashing of the sample over the sides of the sieve. The washing shall be continued until the water coming through the sieve is clear, but in no case shall the washing be continued for more than 30 min. Care shall be taken not to let water accumulate on the No. 200 (74-micron) sieve, because the openings will become clogged and the operation cannot be completed in 30 min. The residues on both sieves shall be dried to constant weight in an atmosphere free from carbon dioxide at a temperature between 212 and 248 F. (100 and 120 C.). The percentage residue retained on each sieve shall be calculated, based on the original weight of the sample. The weight of the material retained on the No. 30 (590-micron) sieve shall be added to the weight of the material retained on the No. 200 (74-micron) sieve to obtain the correct weight of the material retained on the No. 200 (74-micron) sieve.

STANDARD CONSISTENCY OF LIME PUTTY

Apparatus

5. (*a*) *Modified Vicat Apparatus.*—The modified Vicat apparatus shall be used. The apparatus, constructed as shown in Fig. 1, shall consist of a bracket, *A*, bearing a movable brass rod, *B*, 6.3 mm. in diameter and of suitable length to fit the Vicat bracket. A plunger, *C*, 12.5 mm. in diameter, made of aluminum tubing, shall be attached to the lower end of the rod. The total weight of the rod with plunger shall be 30 g. The lower end of the plunger shall be closed without shoulders or curvature and the tube may be loaded with shot to the specified weight. The total weight required may also be obtained by means of a weight, *D*, screwed into the rod. The rod can be held in any position by means of a screw, *E*, and has a mark midway between the end which moves under a scale, *F*, graduated in millimeters, attached to the bracket, *A*.

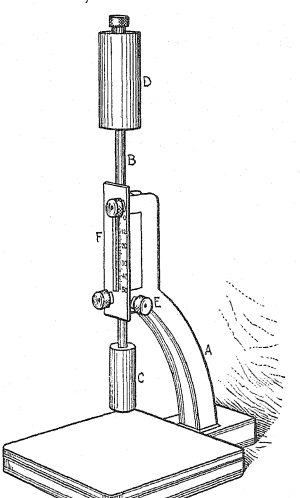


FIG. 1.—Modified Vicat Apparatus.

(*b*) *Mold.*—The conical ring mold shall be made of a noncorroding, nonabsorbent material, and shall have an inside diameter of 7 cm. at the base and 6 cm. at the top, and a height of 4 cm.

(*c*) *Base Plate.*—The base plate for supporting the ring mold shall be of plate glass and about 10 cm. square.

Standard Consistency Determination

6. (*a*) *Procedure.*—Standard consistency of lime putty shall be measured as follows: About 300 g. of hydrated lime

shall be mixed with sufficient water to form a thick putty and stirred with a trowel or spatula for 2 or 3 min. to insure intimate mixing. After aging in a vessel covered with a damp cloth for not less than 16 nor more than 24 hr., this putty shall again be stirred for 2 or 3 min. and a sufficient quantity placed in the ring mold resting on the glass base plate. The mold shall be placed with its larger end resting on the base plate and, after filling, the lime putty shall be struck off flush with the top of the mold. The lime putty, confined in the ring mold resting on the plate, shall then be centered under the rod of the modified Vicat apparatus, Fig. 1. The plunger end, *C*, shall then be brought in contact with the surface of the lime putty and an initial reading taken. The rod shall then be released and the final reading shall be taken 30 sec. after the plunger is released.

(*b*) *Standard Consistency.*—The lime putty shall be of standard consistency when a penetration of 20 mm. is obtained in 30 sec. A permissible variation of plus or minus 5 mm. is allowed

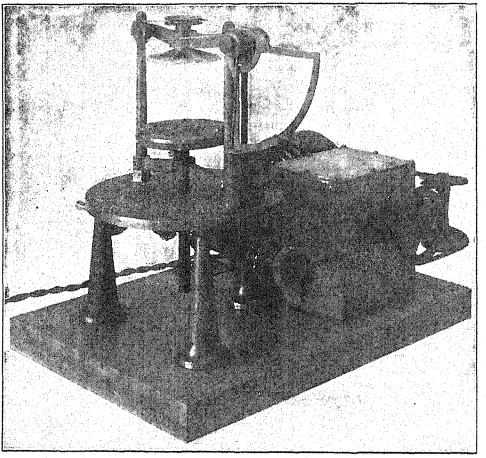


FIG. 2.—Emley Plasticimeter.

Constants of the Machine:

Absorption of porcelain base plate.—Minimum of 40 g. in 24 hr. For rate of absorption of base plates, see Section 9.
Dimension of base plate.—1 in. in thickness by 4 in. in diameter.
Dimensions of disk.— $\frac{1}{32}$ in. in thickness by 3 in. in diameter.
Speed of vertical shaft.—One revolution in 6 min., 40 sec.
Upward movement of base plate.— $\frac{1}{8}$ in. per revolution.
Torque on disk when bob reading is 100.— 14,400 g.cm.

in preparing the sample for the plasticity determination described in Section 7.

(*c*) If the penetration is less than standard, the sample may be removed from the mold, combined with the original aged putty, the whole mixed with more water, stirred for 2 or 3 min., and a suitable quantity retested. If the penetration is more than standard, the sample shall be discarded and a new one prepared.

PLASTICITY OF LIME PUTTY

Plasticity Determination

7. (*a*) The plasticity of lime putty shall be determined by means of the apparatus shown in Fig. 2. A ring mold such as is described in Section 5 (*b*) shall be lubricated with a thin film of water, placed on a porcelain base plate (see Sections 9 and 10), filled with the paste which has been adjusted to standard consistency as described in Section 6, and struck off level. The mold shall be removed by raising it vertically without distorting the paste. The base plate and paste shall be centered in the instrument and the carriage turned up by hand until the surface of the paste is in contact with the disk and the distance between the disk and the top of the base plate is 32 mm. ($1\frac{1}{4}$ in.). The carriage shall then be thrown into gear and the motor started. It is essential that the motor shall be started exactly 120 sec. after the first portion of the paste has been placed in the mold. The time when the first portion of paste is placed in the mold shall be recorded as zero time; the motor is therefore started at 2 min. Care shall be taken to protect the specimen from drafts during the test.

(*b*) The scale reading shall be recorded every minute until the test is completed. The test shall be considered complete when:

(1) the scale reading reaches 100,

(2) any reading is less than the one before, or

(3) the scale reading remains constant for three consecutive readings (2 min.) and the specimen has visibly ruptured or broken loose from the base plate.

The time and the scale reading at the end of the test shall be noted.

Calculation

8. The plasticity figure shall be calculated from the following formula:

$$P = \sqrt{F^2 + (10T)^2}$$

where:

P = plasticity figure,
F = scale reading at the end of the test, and
T = time in minutes from the time when the first portion of paste was put in the mold to the end of the test.

Cleaning and Care of Base Plates

9. In making plasticity determinations much of the success attainable depends upon the condition of base plates. Continued use of the plates without proper cleaning results in clogging of the pores, with reduction in the rate of absorption. After a plate has been used, the excess lime shall be wiped off and the plate immersed in clear water for not less than 2 hr., after which it shall be transferred without drying to a diluted solution of HCl (1:9) where it shall be kept immersed for another 2 hr. It shall then be transferred to a receptacle containing running water for at least 1 hr. The plate is then free of acid and, after the removal of excess water, shall be placed in an oven overnight at a temperature of between 212 and 230 F. (100 and 110 C.) for drying. Before using, the plate shall be cooled to room temperature.

Absorption of Plasticimeter Base Plates

10. (*a*) *Total Absorption.*—Plasticimeter base plates when immersed in water at room temperature for a period of 24 hr. shall absorb not less than 40 g. of water. Before making the determination the plates shall be dried overnight in an oven at a temperature of between 212 and 230 F. (100 and 110 C.) and permitted to cool to room temperature. After immersion and before weighing, the excess water shall be wiped off with a damp cloth.

(*b*) *Rate of Absorption.*[4]—When tested over an area $2\frac{3}{4}$ in. in diameter the water absorbed shall be in accordance with the following:

	Water Absorbed, ml.
First minute	8 to 14
Second minute	5 to $7\frac{1}{2}$
Third minute	4 to $6\frac{1}{2}$
Fourth minute	4 to 6
Fifth minute	$3\frac{1}{2}$ to $5\frac{1}{2}$

SOUNDNESS

Soundness

11. Soundness of hydrated lime shall be determined as follows:

To 20 g. of the sample, 100 g. of standard Ottawa sand shall be added, mixed thoroughly, and enough water added to the mixture to make a good plastic mortar of rather dry consistency. This shall be spread on a clean glass plate to form a layer about $\frac{1}{4}$ in. in thickness by 4 in. square. The pat shall be of even thickness throughout and not tapering at the edges. If the mortar is too dry to work well, more water may be added. The pat shall be placed in a closet for 24 hr. The temperature in the closet shall be between 65 and 75 F. (18 and 24 C.), and there shall be free circulation of air but no direct draft striking the pat. The pat shall then be soaked in water until a film of water stands unabsorbed on the surface of the pat. If the pat has cracked, the consistency of the mortar was too wet and a new pat must be made. Two or three pats made at the same time from mortars of slightly different consistencies will avoid delays.

Twenty grams of the sample shall be mixed with enough water to form a thick cream. This shall be spread in a thin layer on the surface of the pat. It shall be allowed to stand for 15 min. to permit air bubbles to form. It shall be troweled to an even surface, making this skim coat as thin as possible without allowing the sand to show through. The pat shall be put in the closet, under the conditions specified above, for 24 hr. It shall be examined carefully to see that there are no cracks or pops. The pat shall be suspended in a vessel partially filled with cold water, in such a way that the water can boil without touching the pat. The water shall be brought gradually to boiling and kept boiling gently for 5 hr., the pat being surrounded by steam during this time. The water shall then be permitted to cool for at least 12 hr. The pat shall then be removed and examined.

[4] A convenient apparatus for determining the rate of absorption consists of a burette sealed onto an inverted glass funnel from which the stem has been removed. The diameter of the larger end of the funnel shall be ground so as to be $2\frac{3}{4}$ in. in internal diameter. The funnel may be attached to the plate on which the measurement is being made by melted paraffin. The paraffin should not be too hot. A little experience will indicate when it is of the proper consistency.

Tentative Definitions of

TERMS RELATING TO LIME[1]

A.S.T.M. Designation: C 51 – 44 T

ISSUED, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Run-of-Kiln Quicklime.—Quicklime as drawn or discharged from a kiln.

Quicklime Sizes.—Quicklime may be obtained in different sizes depending upon the type of limestone, kind of kiln used, or treatment subsequent to calcining. The sizes commonly recognized are as follows:

(*a*) *Large Lump.*—8 in. and smaller.

(*b*) *Pebble or Crushed.*—2½ in. and smaller.

(*c*) *Ground, Screened, or Granular.*—¾ in. and smaller.

(*d*) *Pulverized.*—Substantially all passing a No. 20 (840-micron) sieve.

Hydraulic Hydrated Lime.—The hydrated dry cementitious product obtained by calcining a limestone containing silica and alumina to a temperature short of incipient fusion so as to form sufficient free calcium oxide to permit hydration and at the same time leaving unhydrated sufficient calcium silicates to give the dry powder its hydraulic properties.

High-Calcium Hydraulic Hydrated Lime.—A hydraulic hydrated lime that contains not more than 5 per cent magnesium oxide (nonvolatile basis).

High-Magnesium Hydraulic Hydrated Lime.—A hydraulic hydrated lime that contains more than 5 per cent magnesium oxide (nonvolative basis).

[1] Under the standardization procedure of the Society, these definitions are under the jurisdiction of the A.S.T.M. Committee C-7 on Lime.

[2] Accepted by the Society at annual meeting, June, 1944.

Tentative Definitions of

TERMS RELATING TO GYPSUM[1]

A.S.T.M. Designation: C 11 – 41 T

ISSUED, 1916; REVISED, 1921, 1925, 1926, 1928, 1929, 1931, 1940, 1941.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Aggregate.—Any inert material used as a filler in stucco, plaster, mortar, concrete, etc., without regard to its function as a binding material.

Consistency.—A property of a material determined by the complete flow-force relation.

Gypsum Concrete.—A combination of aggregate or aggregates with gypsum as a binding medium, which after mixing solidifies into a conglomerate mass.

Gypsum Fiber Concrete.—Gypsum concrete in which the aggregate consists of shavings or chips of wood.

Perforated Gypsum Lath.—A gypsum lath with perforations not less than ¾ in. in diameter, with one perforation for not more than each 16 sq. in. of lath.

Plasticity.—A complex property of a material, involving a combination of the properties of mobility and of yield value.

NOTE.—A plastic material is distinct from a solid material in that it possesses mobility. It is distinct from a fluid material in that it requires a measurable force (yield value) to start flow.

[1] Under the standardization procedure of the Society, these definitions are under the jurisdiction of the A.S.T.M. Committee C-11 on Gypsum.

[2] These definitions, when adopted as standard, will be added to the present Standard Definitions of Terms Relating to Gypsum (A.S.T.M. Designation: C 11 – 41), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Tentative Specifications for

GLAZED MASONRY UNITS[1]

A.S.T.M. Designation: C 126 - 44 T

ISSUED, 1936; REVISED, 1937, 1939, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These specifications cover ceramic glazed[3] brick and ceramic glazed structural clay tile of clay, shale, fire clay or mixtures thereof, with or without the addition of grog or other admixtures. Salt glazed ware is not covered by these specifications. Two grades and two types of glazed masonry units are covered, as follows:

Grade S (select).—For use with mortar joints $\frac{1}{4}$ in. in thickness.

Grade G (ground).—For use where variation of face dimensions must be very small.

Type I (single-faced units).—For general use where only one finished face will be exposed.

Type II (two-faced units).—For use where two opposite finished faces will be exposed.

Basis of Purchase

2. Orders for material under these specifications shall include the following information:

(*a*) *Grade.*—When the grade is not specified, the requirements for grade *S* shall govern.

(*b*) *Type.*—When the type is not specified, the requirements for type I shall govern.

(*c*) *Sizes.*—The sizes shall be specified in accordance with Section 8.

(*d*) *Color and Texture of Finish.*—The color and texture of the finish shall be specified in accordance with Section 7.

(*e*) *Exposure.*—When units are not specified for outdoor exposure, they need not conform to the absorption requirements of Section 5.

(*f*) *Scoring.*—When plaster is to be applied to the backs of the units, scored surfaces shall be specified as prescribed in Section 12.

(*g*) *Coring.*—Unless otherwise specified, either standard or heavy-duty units as prescribed in Section 13 may be furnished.

Description of Units

3. (*a*) *Solid Units.*—Solid units are not perforated.

(*b*) *Multicored Units.*—Multicored units are cored (perforated), the holes or perforations being within the perimeters of the exterior shells and each hole having either a minimum dimension of

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-15 on Manufactured Masonry Units.

[2] Latest revision accepted by the Society at annual meeting, June, 1944.

[3] Ceramic glazed units have a glossy or satin-matt finish of either an opaque or clear glaze, produced by the application of a coating prior to firing and subsequently made vitreous by firing.

½ in. or a cross-sectional area of less than 1 sq. in.

(*c*) *Hollow Units.*—Hollow units contain cells which are hollow spaces enclosed within the perimeter of the exterior shells and having a minimum dimension of not less than ½ in. and a cross-sectional area of not less than 1 sq. in.

Compressive Strength

4. The compressive strengths (based on gross area) of the units shall be not less than the values prescribed in Table I.

TABLE I.—COMPRESSIVE STRENGTHS OF UNITS.

Direction of Coring	Minimum Average for Five Units, psi.	Individual Minimum, One Unit, psi.
Vertical	3 000	2 500
Horizontal	2 000	1 500

Absorption

5. When units are specified by the purchaser for use where exposed to the weather or soil, the average absorption by 24-hr. submersion in cold water shall not exceed 5 per cent and the absorption of any single unit shall not exceed 7 per cent; percentages being based on the original dry weight of the units.

Properties of Finish

6. (*a*) *Imperviousness.*—No stain shall remain on the surface after the imperviousness test, except a slight discoloration in the depressions on matt, stippled, or mottled finishes.

(*b*) *Opacity.*—Discoloration of the body shall not be visible through an opaque glaze in the opacity test. Clear glazes and special decorative glazes shall not be required to meet this requirement.

(*c*) *Resistance to Fading.*—The color of the glaze shall not change in the fading test. Finishes of metallic or special decorative glazes shall not be required to meet this requirement.

(*d*) *Resistance to Crazing.*—The glaze shall not craze, spall, or crack when subjected to one cycle of autoclaving in the crazing test.

Color and Texture

7. The color and texture of the finished surfaces of the units shall be as specified and shall conform to an approved reference sample. The reference sample shall consist of five stretcher units representing the ranges of shade and texture.

TABLE II.—SIZES OF SINGLE-FACED UNITS.

Standard Face Dimensions		Standard Thicknesses, in.
Height, in.	Length, in.	
2¼	8	1¾ or 3⅝
3⅝	8	1¾ or 3⅝
5	8	1¾, 3⅝, 5⅝ or 8
5	12	1¾, 3⅝, 5⅝ or 8
8	12	1¾ or 3⅝
8	16¾	1¾ or 3⅝

Sizes

8. The units shall be of the sizes and shapes specified (Note).

NOTE.—The sizes shown in Table II are standard in the industry for single-faced units (type I). Two-faced units (type II) of the same face dimensions as units of type I are standard in the industry in 3⅝-in. thickness only.

Number of Cells

9. (*a*) Units may be solid, multicored, or hollow. Requirements as to number of cells shall apply to hollow units only (Section 3 (*c*)). Hollow units of 6-in. and 8-in. thickness shall have not less than two cells or rows of cells across the thickness of the unit.

(*b*) Double-shell tile shall be considered as having one additional cell across the thickness of the unit if either:

(*1*) The width of the voids within each of the two double shells of the tile is not less than $\frac{1}{4}$ in., or

(*2*) The combined thickness of the inner and outer shells on each side of the tile is not less than 1 in.

(*c*) The face shells of single-shell tile with multicored or solid face shells at least $1\frac{1}{2}$ in. in thickness on both sides of the tile shall be considered as one additional cell in wall thickness, provided the volume of the holes in multicored shells does not exceed 35 per cent of the gross volume of the face shell and the minimum distance from the perimeter of any hole to either side of the shell is not less than $\frac{3}{8}$ in.

TABLE III.—TOLERANCES ON FACE DIMENSIONS.

Standard Face Dimension (Height or Length), in.	Maximum Permissible Variation, plus or minus, from Standard Face Dimension, in.		Maximum Permissible Variation in Face Dimension, Largest to Smallest of Units in One Lot[a] in.	
	Grade S	Grade G	Grade S	Grade G
$2\frac{1}{4}$	$\frac{1}{16}$	...	$\frac{3}{32}$	...
$3\frac{1}{2}$	$\frac{5}{32}$	$\frac{1}{8}$	$\frac{3}{32}$	$\frac{1}{16}$
5	$\frac{3}{32}$	$\frac{1}{16}$	$\frac{1}{8}$	$\frac{1}{16}$
8	$\frac{1}{8}$	$\frac{1}{16}$	$\frac{5}{32}$	$\frac{1}{16}$
12	$\frac{5}{32}$	$\frac{1}{16}$	$\frac{1}{4}$	$\frac{1}{16}$
$16\frac{1}{4}$	$\frac{1}{4}$	$\frac{1}{16}$	$\frac{1}{4}$	$\frac{1}{16}$

[a] Size of lot shall be determined by agreement between the purchaser and the seller.

Shell and Web Thickness

10. (*a*) The minimum distance from the perimeter of any hole to the outer surface of the shell of multicored units shall be not less than $\frac{3}{4}$ in.

(*b*) The average over-all thickness of the shells of vertical cell hollow units, measured between the inner and extreme outer surfaces, shall be not less than $\frac{3}{4}$ in. The thickness of webs shall be not less than $\frac{1}{2}$ in.

(*c*) The average over-all thickness of the face shells of horizontal cell hollow units, measured between the inner and extreme outer surfaces, shall be not less than $\frac{3}{4}$ in. The least thickness of the other shells of horizontal cell hollow units and the thickness of the webs shall be not less than $\frac{1}{2}$ in.

(*d*) The horizontal width of any cell in horizontal cell hollow units shall not exceed $4\frac{1}{2}$ times the average over-all thickness of either the upper or lower bearing shells.

Tolerances on Dimensions

11. (*a*) The dimensions of the faces of units of types I and II shall not differ from those specified by more than the amounts given in Table III.

(*b*) The thicknesses (measured perpendicular to face of wall as laid) of units of grades *S* and *G* shall not differ from those specified by more than the amounts given in Table IV.

TABLE IV.—TOLERANCES ON THICKNESS OF UNITS.

Standard Thickness, in.	Maximum Permissible Variation, plus or minus, from Standard Thickness, in.		Maximum Permissible Variation in Thickness Largest to Smallest of Units in One Lot,[a] in.	
	Type I	Type II	Type I	Type II
$1\frac{3}{4}$	$\frac{1}{8}$	...	$\frac{1}{8}$	...
$3\frac{5}{8}$	$\frac{1}{8}$	$\frac{1}{8}$	$\frac{1}{8}$	$\frac{1}{8}$
$5\frac{5}{8}$	$\frac{5}{16}$	...	$\frac{1}{4}$	...
8	$\frac{1}{4}$	...	$\frac{5}{16}$	...

[a] Size of lot shall be determined by agreement between the purchaser and the seller.

(*c*) Distortions of the face or of the edge of a face from a straight line, when measured as the maximum ordinate from the line to the face or the edge, shall not exceed the amounts given in Table V.

(*d*) Tolerances for units having dimensions not listed in Tables III, IV, and V shall be the same as for the next larger standard dimension.

Scoring

12. When scored-back units are specified, each groove shall be not less than $\frac{1}{8}$ nor more than $\frac{1}{4}$ in. in depth, nor more than 1 in. in width. The area covered

by the grooves shall not exceed 50 per cent of the area of the scored faces.

Coring

13. When heavy-duty units are specified, the units may be either solid or cored. If cored, the volume of the holes shall not exceed the values given in Table VI.

Workmanship

14. (*a*) The body of the units shall be free from cracks or other imperfections which would impair the strength or durability of the masonry.

TABLE V.—PERMISSIBLE DISTORTIONS OF FACE AND EDGE OF FACE.

Standard Face Dimensions, in.	Maximum Ordinate From a Straight Line, in.	
	Grade S	Grade G
2¼ by 8	1/32	...
2⅝ by 8	1/16	1/16
5 by 8	1/16	1/16
5 by 12	1/32	1/32
8 by 12	1/32	1/16
8 by 16¾	1/16	1/32

TABLE VI.—CORING OF HEAVY-DUTY UNITS.

Standard Thickness of Unit, in.	Maximum Coring, per cent[a]
3⅞	25
5⅝	35
8	35

[a] Calculated as 100 times the volume of the holes made by coring divided by the gross volume of the units.

(*b*) The finished face (one face) of stretcher units and the finished faces of shapes that will be exposed when in place shall be covered with a ceramic glaze (opaque or clear) of uniform quality. The glaze shall be free from chips, crazes, blisters, crawling, or other imperfections visible at a distance of 5 ft.

Inspection

15. Facilities shall be provided the purchaser for sampling and inspection at either the place of manufacture or the site of the construction, as specified by the purchaser. At least ten days from the time of sampling shall be allowed for completion of the tests.

Test Specimens

16. Specimens of whole units shall be selected by the purchaser or his representatives at the place or places designated in the purchase order. Eight stretcher units shall be tested for a lot of 10,000 units or fraction thereof; for larger lots, five additional specimens shall be tested for each 30,000 units or fraction thereof. Other representative samples may be taken at any time or place at the discretion of the purchaser. When less than 1,000 units of the same size, shape and finish are ordered, tests of these units shall not be required.

Methods of Test

17. (*a*) *Compressive Strength Test.*— Compressive strength tests shall be made on three specimens in accordance with Sections 9, 10, 11, and 12 of the Standard Methods of Sampling and Testing Brick (A.S.T.M. Designation: C 67).[4] Specimens used in the crazing test (Paragraph (*f*)) shall not be used in the compressive strength test. When the same three specimens are to be subjected to both absorption and compressive strength tests, they shall be dried to constant weight before subjecting them to the compression test.

(*b*) *Absorption Test.* — The absorption test shall be made on three whole specimens or on three pieces cut or sawed from the three specimens, each containing not less than 16 sq. in. of glazed surface and of the thickness of the body. When the same three specimens are to be subjected to both absorption and crazing tests (Paragraph (*f*)), the absorption test shall be made first. The absorption test shall be made in

[4] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

accordance with Sections 15 and 16 of A.S.T.M. Methods C 67.

(*c*) *Imperviousness Test.*—Blue-black fountain pen ink shall be applied liberally to the glazed surface and allowed to remain for 5 min. The surface shall then be washed with a wet cloth and running water, and examined for staining of the finish.

(*d*) *Opacity Test.*—Blue-black fountain pen ink shall be applied liberally to the body along a 2-in. length of the edge of the finished face. After 5 min., the finish shall be examined for opacity. When the same specimens are to be subjected to both opacity and crazing tests (Paragraph (*f*)), the opacity test shall be made first.

(*e*) *Fading Test.*—A portion of the glazed surface of two of the specimens shall be submerged in a 10 per cent solution of hydrochloric acid for 3 hr. The solution shall be maintained at a temperature of 60 to 80 F. (15 to 27 C.). The finishes shall then be rinsed, dried, and examined for change of color.

(*f*) *Crazing Test.*—The crazing test shall be made on three whole dry units previously tested for imperviousness of finish (Paragraph (*a*)). Specimens subjected to the fading test (Paragraph (*e*)) shall not be used. The autoclave shall be of sufficient capacity to contain all the specimens of the same texture, color, and size. The apparatus shall be equipped with a safety valve, a blowoff valve, a thermometer, and a pressure gage accurate within 2 per cent of the scale range, and a heater or other means for creating and maintaining steam pressure within the autoclave. The specimens shall be placed above the water in the autoclave at room temperature. After fastening the autoclave head in place, the water in the bottom shall be heated or saturated steam shall be admitted slowly. The blowoff valve shall be kept open until steam escapes and most of the air has been expelled. After closing the blowoff valve, the steam pressure shall be increased at such a rate that it reaches 150 psi. in $\frac{3}{4}$ to 1 hr. A constant pressure of 150 $\pm$ 5 psi. with saturated steam shall be maintained for an additional hour. The blowoff valve shall then be opened slightly and the steam allowed to escape at a rate to be fully released in from 15 to 30 min. The autoclave head shall be loosened, but not removed, and the specimens permitted to cool gradually to a temperature not exceeding 120 F. in a period not less than 3 hr. The specimens shall then be removed and blue-black fountain pen ink rubbed upon the glazed surfaces to aid in the detection and examination of failures.

Tentative Specifications for

VITRIFIED CLAY FILTER BLOCK FOR TRICKLING FILTERS[1]

A.S.T.M. Designation: C 159 – 41 T

ISSUED, 1941.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. (*a*) These specifications cover vitrified clay filter block made from clay or shale or mixtures thereof. Two types of filter block are covered, as follows:

Type I.—A one-piece filter block suitable for use in constructing a single-course trickling filter floor, which provides continuous drainage channels or ducts through the lower portion of the block for the conveyance of liquids from the filter bed and drainage and aeration grilles in the upper portion of the block for the passage of liquids from, and air into the filtering media.

Type II.—A two-piece filter block suitable for use in constructing a two-course trickling filter floor, which provides drainage and aeration. When placed or assembled, a course of the lower pieces or units forms continuous drainage channels or ducts for the conveyance of liquids from the filter bed and for the passage of air into the filter bed. An assembly of the upper units forms drainage and aeration grilles for the passage of liquids from, and air into the filtering media.

(*b*) The purchaser should specify the type or types of block desired.

Compressive Strength

2. The average compressive strength of five filter block shall be not less than 600 psi., based on the gross area of the block, with the load applied in the same direction as in service. No individual block shall have a compressive strength of less than 500 psi.

Absorption

3. The average water absorption of five filter block, by 1-hr. submersion in boiling water, shall not exceed 6 per cent of the dry weight of the block.

Shape

4. (*a*) Type I filter block shall be of the self-spacing type and shall be rectangular in plan, as laid. When placed

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-15 on Manufactured Masonry Units.

[2] Accepted by the Society at annual meeting, June, 1941.

in parallel rows on the subfloor of the filter bed, the cells in a single course of type I filter block shall form continuous drainage ducts, and the apertures or slots shall provide for drainage and aeration of the filter media.

(*b*) Both the units for the lower or drainage course and the units for the upper or aeration course of type II filter block shall be of the self-spacing type and shall be rectangular in plan, as laid. When placed in parallel rows on the subfloor of the filter bed, the lower units shall form continuous drainage channels. The upper units, when laid in parallel rows on the lower units, shall provide for drainage and aeration of the filter media.

Permissible Variations in Dimensions

5. The length, width, or height of a filter block shall not vary by more than $\frac{1}{4}$ in. per ft. over or under the nominal dimension.

Apertures

6. (*a*) Apertures in the top of type I filter block shall be not more than $1\frac{1}{2}$ in. in width, but may vary in length. The aperture area shall be not less than 20 per cent of the top area of the filter block when in position of use.

(*b*) Apertures in the top or aeration course of type II filter block shall be not more than $1\frac{1}{8}$ in. in width, but may vary in length. The aperture area shall be not less than 40 per cent of the top area of the block when in position of use.

Shell and Web Thickness

7. (*a*) The exterior walls (shells) of type I filter block shall be not less than $\frac{9}{16}$ in. in thickness and the interior webs shall be not less than $\frac{1}{2}$ in. in thickness.

(*b*) The exterior walls (shells) and the interior webs and struts of the type II filter block comprising the lower or drainage course shall be not less than $\frac{9}{16}$ in. in thickness. The exterior walls (shells) and the longitudinal webs of the type II filter block comprising the top or aeration course shall be not less than $1\frac{1}{2}$ in. in thickness. Stiffening webs, for stiffening the longitudinal webs, shall be not less than $\frac{9}{16}$ in. in thickness.

Workmanship and Finish

8. (*a*) Filter block may be either glazed or not glazed, unless otherwise specified in the purchase order.

(*b*) The lower bearing surface of filter block shall not vary from a plane by more than $\frac{1}{8}$ in. per ft. of the greatest length or width of the block.

(*c*) All filter block shall be well burned and substantially free of laminations and fractures. All block shall be free of cracks exceeding 2 in. in length, of chips exceeding 2 by 1 by $\frac{3}{16}$ in. in dimensions, and of blisters exceeding 2 in. in diameter and projecting more than $\frac{1}{8}$ in. above the normal surface.

Marking

9. All filter block shall bear the name, initials, or trade mark of the manufacturer. These marks shall be indented on the exterior of the block and shall be legible.

Rejection

10. If the test specimens fail to conform to the requirements for compressive strength or absorption, the manufacturer may sort the shipment, and new samples shall be selected by the purchaser from the retained lot and tested at the expense of the manufacturer. In case the second set of test specimens fails to meet the requirements, the entire lot shall be rejected.

Expense of Tests

11. Except as specified in Section 10, the expense of inspection and testing shall be borne by the purchaser.

Sampling and Testing

12. (*a*) The purchaser or his authorized representative shall be accorded proper facilities for sampling and inspection of the filter block, both at the place of manufacture and at the site of the work. At least ten days from the time of sampling should be allowed for completion of the tests.

(*b*) Filter block shall be sampled and tested in accordance with the Standard Methods of Sampling and Testing Structural Clay Tile (A.S.T.M. Designation: C 112) of the American Society for Testing Materials.[3]

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[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Tentative Method of Test for

COMPRESSIVE STRENGTH OF NATURAL BUILDING STONE[1]

A.S.T.M. Designation: C 170 – 41 T

ISSUED, 1941.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. This method covers the sampling, preparation of specimens, and procedure for determining the compressive strength of natural building stone.

Sampling

2. The sample shall be selected to represent a true average of the type or grade of stone under consideration and shall be of the quality supplied to the market in finished form under the type designation to be tested. The sample may be selected by the purchaser or his authorized representative from quarried stone or taken from the natural ledge and shall be of adequate size to permit the preparation of the desired number of test specimens. When perceptible variations occur, the purchaser may select as many samples as are necessary for determining the variation in compressive strength.

Apparatus

3. (*a*) Any testing machine conforming to the Standard Methods of Verification of Testing Machines (A.S.T.M. Designation: E 4) of the American Society for Testing Materials[3] and to the speed of testing requirements prescribed in Section 6 of these methods may be used.

(*b*) In vertical testing machines, the spherical bearing block shall be suspended from the upper head of the machine in such a manner that the contact plate remains in a central position (spherical surfaces in full contact) when not loaded. The spherical surfaces shall be well lubricated, and the center of curvature shall lie in the surface of contact with the specimen.

Test Specimens

4. (*a*) The test specimens may be cubes, square prisms, or cylinders and shall be cut from the sample with saws or core drills. The diameter or lateral dimension (distance between opposite vertical

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-18 on Natural Building Stones.

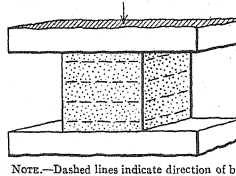
[2] Accepted by Committee E-10 on Standards, October 23, 1941.

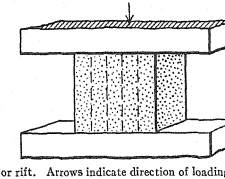
[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

faces) shall be not less than 2 in. (Note 1), and the ratio of height (Note 2) to diameter or lateral dimension shall be not less than 1:1. At least three specimens shall be prepared for each condition of the test; that is, when the compressive strength is desired for the wet and dry conditions but in only one direction, such as perpendicular to the bed (or rift) (see Fig. 1 (*a*)), six specimens will be required. For wet and dry strength tests both perpendicular and parallel to the bed (or rift) (see Fig. 1 (*a*) and (*b*)), twelve specimens are required (Note 3). The load-bearing faces shall be finished by grinding to as nearly true and parallel planes (Note 4) as practicable.

(*b*) The load-bearing surfaces and the direction of bedding (or rift) shall be marked on each specimen after finishing.

(*c*) The load-bearing areas of the specimen shall be calculated from measurements taken midway between the load-bearing surfaces. The dimensions of the specimens shall be measured to the nearest 0.02 in., and the load-bearing areas calculated to the nearest 0.04 sq. in.





NOTE.—Dashed lines indicate direction of bedding or rift. Arrows indicate direction of loading.
(*a*) Loading Perpendicular to Bedding or Rift. (*b*) Loading Parallel to Bedding or Rift.

FIG. 1.—Methods of Applying Load with Reference to Bedding or Rift.

Conditioning

5. (*a*) *Dry Condition.*—Specimens to be tested in the dry condition shall be dried at 105 ± 2 C. for 24 hr.

(*b*) *Wet Condition.*—Specimens to be tested in the wet condition shall be immersed in water at 20 ± 5 C. for 48 hr. and tested immediately after removal from the water bath.

Procedure

6. The specimens shall be centered in the testing machine and the initial load applied at a rate that will permit hand adjustment of the contact plate on the specimen. The plate should be rotated back and forth through an angle of about 30 deg. under a small load to properly seat the spherical block, but care shall be taken not to move the specimen out of the central position. Preferably, the rate of loading should not exceed 100 psi. per sec., but this requirement may be considered as being met if the speed of the loading head is not more than 0.05 in. per min.

Calculations

7. (*a*) The compressive strength of each specimen shall be calculated as follows:

$$C = \frac{W}{A}$$

where:

C = compressive strength of the specimen, in pounds per square inch,

W = total load in pounds on the specimen at failure, and

A = calculated area of the bearing surface in square inches.

Each individual result shall be rounded off to the nearest 100 psi.

(*b*) When the ratio of height to diameter or lateral dimension is greater than 1:1, the results shall be calculated to the equivalent for the 1:1 ratio, as follows:

$$C_c = \frac{C_p}{0.778 + 0.222(b \div h)}$$

where:

C_c = compressive strength of an equivalent cubical specimen,

C_p = compressive strength of the specimen having a height greater than the diameter or lateral dimension,

b = diameter or lateral dimension, and

h = height.

Report

8. (*a*) The average compressive strength of all specimens loaded as shown in Fig. 1 (*a*) shall be reported as the compressive strength perpendicular to the bedding (or rift), and the average compressive strength of all specimens loaded as shown in Fig. 1 (*b*) shall be reported as the compressive strength parallel to the bedding (or rift).

(*b*) The following additional information shall be reported:

(*1*) Identification of the sample, including name and location of the quarry, name or position of the ledge, date when sample was taken and trade name or grade of stone,

(*2*) Size and shape of specimens used in the tests, and

(*3*) A description of the way in which the specimens were prepared.

EXPLANATORY NOTES

NOTE 1.—For very coarse-grain materials like some of the granites, the diameter of the specimen should not be less than 2.5 in.

NOTE 2.—The height of the specimen is considered as the distance between the load-bearing faces.

NOTE 3.—In some materials, such as granite, three directions with respect to fissility are recognized, as follows: "rift" (the plane of easiest splitting), "grain" (the plane of next easiest splitting), and "head-grain" (the plane of hardest splitting). Occasionally, tests are required for determining the strength perpendicular to each of these directions. In such cases, the sample shall be marked at the quarry to show which faces are grain, rift, and head-grain, and the required number of specimens shall be prepared with load-bearing faces parallel to each of these planes and properly labeled for the various tests.

NOTE 4.—Accuracy of test results depends largely on uniform distribution of the load over the bearing faces. In order to grind the surfaces to reasonably true planes, considerable care is necessary. The following procedure is suggested: Assuming that the specimen is a rectangular prism and the load is to be applied to the ends, mark two adjacent sides for reference, then grind the ends on a grinding wheel or lap until they are perpendicular to these reference sides as gaged by a try square. Complete the grinding by rubbing the ends on a smooth machine-planed surface of a cast iron plate with No. 80 emery and water. The specimen should be grasped as near the surface of the plate as possible to prevent rocking of the specimen. A suitable way to determine when the surfaces are reasonably plane is to dip the specimen in water and press the ends on a smooth machine-planed and polished surface of a 10-lb. weight. If the weight can be lifted by raising the specimen, the surfaces may be considered to be sufficiently accurate.

Tentative Specifications for

MORTAR FOR REINFORCED BRICK MASONRY[1]

A.S.T.M. Designation: C 161 - 44 T

ISSUED, 1941; REVISED, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. (*a*) These specifications cover mortar for use in the construction of reinforced brick masonry structures. Two alternative specifications are covered, as follows:

Property Specifications in which the acceptability of the mortar is based on the properties of the ingredients (materials) and the properties (water retention and compressive strength) of the mortar mixture (Sections 2 to 7, inclusive, and Section 10).

Proportion Specifications in which the acceptability of the mortar is based on the properties of the ingredients (materials) and a definite composition of the mortar consisting of fixed proportions of the ingredients (Sections 8 to 10, inclusive).

(*b*) The purchaser shall indicate under which of these specifications the mortar will be accepted.

PROPERTY SPECIFICATIONS

Materials

2. (*a*) Materials used as ingredients in the mortar shall conform to the requirements specified in the following Paragraphs (*b*) to (*h*):

(*b*) *Cementitious Materials.*—Cementitious materials shall conform to one of the following specifications of the American Society for Testing Materials, as specified:

Portland Cement.—Type I or type III of the Standard Specifications for Portland Cement (A.S.T.M. Designation: C 150).[3]

Masonry Cement.—Tentative Specifications for Masonry Cement (A.S.T.M. Designation: C 91).[3]

Quicklime.—Standard Specifications for Quicklime for Structural Purposes (A.S.T.M. Designation: C 5).[3]

Hydrated Lime.—Standard Specifications for Normal Finishing Hydrated Lime (A.S.T.M. Designation: C 6).[3]

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-12 on Mortars for Unit Masonry.

[2] Revision accepted by the Society at annual meeting, June, 1944.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(*c*) *Aggregates.*—Aggregates shall conform to the Standard Specifications for Aggregate for Masonry Mortar (A.S.T.M. Designation: C 144).[3]

(*d*) *Water.*—Water shall be clean and free of deleterious amounts of acids, alkalies, or organic materials.

(*e*) *Admixtures.*—Integral waterproofing compounds, accelerators, or other admixtures not definitely mentioned in the specifications shall not be used in mortar for use in reinforced brick masonry without approval in writing from the purchaser.

(*f*) *Mortar Colors.*—Only pure mineral mortar colors shall be used. The brand and quality of such coloring, and the amount to be used shall, unless definitely stipulated in the specifications, be approved in writing by the purchaser.

(*g*) *Anti-freeze Compounds.*—No anti-freeze liquid, salts or other substances shall be used in the mortar to lower the freezing point.

(*h*) *Storage of Materials.*—Cementitious materials and aggregates shall be stored in such a manner as to prevent deterioration or intrusion of foreign material. Any material that has become unsuitable for good construction shall not be used.

Mortar and Grout

3. (*a*) Mortar shall consist of a mixture of cementitious material and aggregate conforming to the requirements specified in Section 2, to which sufficient water has been added to bring the mixture to a plastic state. The volume of aggregate in mortar shall be at least 2.4 times but not more than 3 times the volume of cementitious materials.

(*b*) Grout shall consist of mortar into which sufficient additional water has been incorporated to cause the mixture to flow readily.

Water Retention

4. Mortar and grout after suction for 60 sec. shall have a flow greater than 65 per cent of the flow immediately after mixing.

Compressive Strength

5. The average compressive strength of three 2-in. cubes of mortar or grout (before thinning) shall be not less than 1600 psi. at the age of 7 days and 2500 psi. at the age of 28 days.

Sampling

6. Water retention and compressive strength of mortar shall be determined on mortar mixed in the laboratory of similar ingredients to those used in the construction. The proportions, except water, shall be the same as those that will be used in the mortar as mixed on the job.

Methods of Testing

7. Water retentivity and compressive strength of mortar shall be determined in accordance with the test procedures described in the Tentative Specifications for Masonry Cement (A.S.T.M. Designation: C 91),[3] with the exception that the mortar sample mixed in the laboratory shall be made using the same proportions (except water), and the same ingredients (cementitious materials and aggregate) that will be used for the mortar in the construction.

Proportion Specifications

Materials

8. Materials used as ingredients in the mortar shall conform to the requirements of Section 2 and, in addition, to the following requirements:

(*a*) *Masonry Cement.*—The average compressive strength of not less than three 2-in. mortar cubes, molded and

tested in accordance with the procedure described in the Tentative Specifications for Masonry Cement (A.S.T.M. Designation: C 91),[3] shall be not less than 2500 psi. at 28 days.

(*b*) *Hydrated Lime.*—Hydrated lime shall conform to either of the two following requirements:

(*1*) The total free (unhydrated) calcium oxide (CaO) and magnesium oxide (MgO) shall be not more than 8 per cent by weight (calculated on the as-received basis for hydrates).

(*2*) When the hydrated lime is mixed with portland cement in the proportion specified in Section 9 (*a*), the mixture shall give an autoclave expansion of not more than 0.50 per cent when tested in accordance with the Standard Method of Test for Autoclave Expansion of Portland Cement (A.S.T.M. Designation: C 151).[3]

Hydrated lime intended for use when mixed dry with other mortar ingredients shall have a plasticity figure of not less than 200 when tested 15 min. after adding water. The test otherwise shall be made in accordance with Sections 7 to 10, inclusive, of the Tentative Methods of Physical Testing of Quicklime and Hydrated Lime (A.S.T.M. Designation: C 110).[3]

(*c*) *Lime Putty.*—Lime putty made from either quicklime or hydrated lime shall be soaked for a period sufficient to produce a plasticity figure of not less than 200 and shall conform to either the requirements for limitation on total free oxides of calcium and magnesium or the autoclave test specified for hydrated lime in Paragraph (*b*).

Mortar and Grout

9. (*a*) Mortar shall consist of a mixture of cementitious materials and aggregate conforming to the requirements specified in Section 8, mixed in one of the following proportions to which sufficient water has been added to reduce the mixture to a plastic state:

MORTAR PROPORTIONS BY WEIGHT

Cement	Hydrated Lime	Aggregate, Dry
1 bag portland cement	10 lb.	240 lb.
1 bag masonry cement	...	240 lb.

MORTAR PROPORTIONS BY VOLUME

Cement	Hydrated Lime or Lime Putty	Aggregate, Damp and Loose
1 cu. ft. portland cement	0.25 cu. ft.	3 cu. ft.
1 cu. ft. masonry cement	...	3 cu. ft.

NOTE.—Volume proportions shall be based on the following weights per cubic foot:

	Weight, lb. per cu. ft.
Portland cement	94
Masonry cement	weight printed on bag
Hydrated lime	40
Sand, damp and loose	1 cu. ft. contains 80 lb. of dry sand

GENERAL PROVISIONS APPLICABLE TO BOTH SPECIFICATIONS

Costs of Tests

10. Unless otherwise specified in the purchase order, the costs of tests shall be borne as follows:

(*a*) If the results of the tests show that the mortar does not conform to the requirements of these specifications, the costs shall be borne by the seller.

(*b*) If the results of the tests show that the mortar does conform to the requirements of these specifications, the costs shall be borne by the purchaser.

Tentative Method of Test for

FIRE-RETARDANT PROPERTIES OF WOOD[1]

A.S.T.M. Designation: C 160 – 41 T

ISSUED, 1941.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. This method of test covers the procedure for fire tests applicable to untreated wood and wood chemically treated by impregnation to reduce flammability for permanent use in construction. Requirements for performance under this test shall be as prescribed in the specifications applicable to the materials intended for specific uses.

Sampling

2. (*a*) The shipment shall be carefully examined and a representative sample selected. One sample shall be taken from each 5000 board feet or fraction thereof. Different species, and pieces of the same species with widely different thicknesses, shall be considered as separate lots and sampled accordingly. Each sample shall consist of a number of individual pieces selected so as to include material from different sections of a finished unit or different lengths of finished trim, or to include variations such as rate of growth, density, and other visible nonuniformities. Samples shall be cut not closer than 2 ft. from the ends of treated boards.

(*b*) A composite test specimen shall be prepared from each sample, unless otherwise specified by the purchaser. Individual pieces sampled from one lot shall be marked for identification and tested separately. Further requirements as to location of samples and number of samples may be applied for materials intended for specific uses as prescribed in the specifications.

(*c*) Unless otherwise specified by the purchaser or jurisdictional authority, the sampling shall be done at the point of delivery.

Apparatus

3. The apparatus shall consist of the following:

(*a*) *Test Chamber.*—A test chamber which may be either a room free from noticeable drafts, or a fume hood. If a fume hood is used, the ventilation shall be such as to cause a minimum movement of air to remove the smoke or gases.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-5 on Fire Tests of Materials and Construction.

[2] Accepted by the Society at annual meeting, June, 1941.

(*b*) *Wire Frame and Ring Stand.*—A wire frame (see Fig. 1) suitable for supporting the specimen stacked in the form of a crib, and a ring stand 6 in. in inside diameter. The wire frame shall consist of four vertical wires, with a clear spacing of 2 in. to form a cage with square sections, held in position at the top by transverse wires on each side, and provided with transverse wires near the bottom which serve as spacers and supports for the specimen. All wire

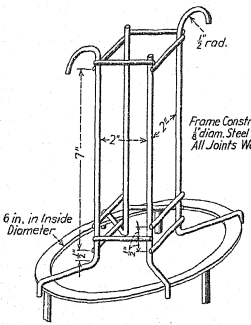


FIG. 1.—Wire Frame and Ring Stand for Supporting Crib Specimen.

used in the frame shall be steel wire ⅛ in. in diameter and connections shall be made by welding. Dimensions of the wire frame and the manner in which it is supported on the ring stand shall be as shown in Fig. 1. The frame shall be vertical and the hooks at the ends of the supporting arms made so that the frame will automatically be centered for any placement, with a minimum clearance to permit removal from the ring. These hooks serve as a self-centering device.

(*c*) *Burner.*—A Meker burner with perforated grid 2 in. in diameter.

(*d*) *Flame Shield.*—A flame shield (Fig. 2) for calibrating and checking the burner flame. The shield shall consist of a cylindrical section, 8 in. in diameter and 12 in. in height, and a conical top with an opening 2¾ in. in diameter. A No. 18-gage chromel-alumel thermocouple shall be mounted with its junction ½ in. over the center of the top opening of the shield, for calibration of the burner flame. The shield shall be

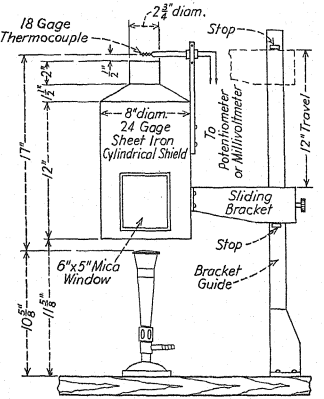


FIG. 2.—Flame Shield.

made of No. 24-gage sheet iron and shall be supported so that when the burner is placed directly under the center of the shield, the distance from the top of the burner grid to the thermocouple is 17 in. (see Fig. 2).

(*e*) *Balance.*—A balance accurate at least to 0.1 g. and provided with a suitable scale pan.

(*f*) *Timer.*—A timing device recording minutes and seconds.

(*g*) *Oven.*—A thermostatically-controlled drying oven.

(*h*) *Meter.*—A potentiometer or millivoltmeter for indicating thermocouple emf. or temperature.

Test Specimens

4. (*a*) The specimen shall consist of 24 pieces $\frac{1}{2}$ by $\frac{1}{2}$ in. in cross-section and 3 in. in length, with surfaces smooth-sawed to dimensions within plus or minus $\frac{1}{32}$ in. The specimen shall be representative of the entire cross-section of the sample.

(*b*) For samples taken from boards having thicknesses ranging from $\frac{1}{2}$ to $1\frac{1}{8}$ in., the composite specimen shall be prepared by cutting parallel $\frac{1}{2}$ by $\frac{1}{2}$-in. pieces longitudinally from one or both faces across the full width of the board. For samples taken from trim of small sectional area, the specimen pieces will ordinarily be suitable as cut by any convenient sawing operation.

(*c*) For material of greater thickness, pieces shall be cut from one face and the center of the section, across the full width.

(*d*) When the material to be tested is less than $\frac{1}{2}$ in. in thickness, the specimen pieces may be built up in laminated form with plies of approximately equal thickness. The plies shall be dressed smooth and assembled by nailing with $\frac{1}{2}$-in. steel wire brads (No. 20 B.w.g., 0.035-in. nominal thickness) driven obliquely, spaced 1 in. apart, and symmetrically located on the 3-in. length. The laminated pieces should preferably be prepared in longer lengths and greater width to permit of cutting to the final specimen dimensions with smooth-sawed faces on the laminated edges. The weight of nails used shall be recorded for correction of the weight of specimen.

(*e*) A composite specimen prepared from more than one sample piece shall be taken so as to be representative of the average quality of the materials to be tested.

Conditioning of Test Specimens

5. (*a*) The moisture content of the specimen when tested shall be 7 ± 3 per cent by weight of dry material. The moisture determination shall be made on a specimen approximately $\frac{3}{4}$ in. in thickness cut transversely at an intermediate section of the sample, or on a moisture specimen consisting of at least four additional $\frac{1}{2}$ by $\frac{1}{2}$ by 3-in. pieces of the prepared test specimen. The moisture specimen shall be weighed to 0.1 g. or less and dried for 24 hr. at 212 ± 3 F. The weight of the test specimen shall be recorded at the time of weighing the moisture specimen. If the moisture content exceeds 10 per cent, the sample or prepared specimen shall be conditioned by drying at temperatures not exceeding 150 F. until the moisture content is within the prescribed limits. Low moisture content can be increased by storage in air on a screen over a pan of water. If not tested immediately, the specimen shall be stored, after conditioning, in such a manner as to prevent absorption or loss of water.

(*b*) The weight to which the sample must be conditioned to contain 7 per cent moisture shall be calculated as follows:

$$W_t = \frac{1.07}{1 + X} \times W_o$$

where:

W_t = desired weight of sample containing 7 per cent moisture at time of test,

W_o = original weight of sample before conditioning, and

X = moisture content of sample.

NOTE: *Example.*—If $X = 15$ per cent, the sample would be dried to a weight of 0.93 W_o to contain 7 per cent moisture.

Procedure

6. (*a*) The specimen pieces shall be placed in the wire frame in 12 tiers of two pieces each, 1 in. apart, with the pieces in each tier placed perpendicular to those in the tier below, thus forming a crib (see Fig. 3). The pieces shall be taken from the composite specimen at random and stacked without regard for original position in the sample, unless otherwise specified. The first tier shall be placed on the lower wires of the frame. The first three tiers will then be supported by wires and the remaining nine tiers built up. The spacing of the pieces shall be duplicated by placing each piece in contact with the vertical wires of the frame, permitting approximately equal overhang at the ends as determined by visual inspection. The frame with the stacked crib in place shall be centered on the ring stand. If tests are made in an open room, the flame shield shall be placed around the specimen during the test. If tests are made in a fume hood with draft controlled as specified in Section 3 (*a*), the shield need not be in place around the specimen during the test.

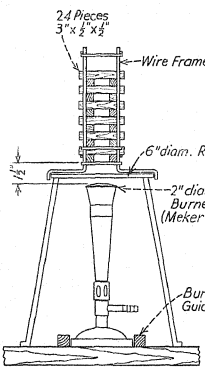


FIG. 3.—Crib Test Assembly.

(*b*) The Meker burner shall be adjusted to produce a blue flame $10 \pm \frac{1}{2}$ in. in height. The air adjustment of the burner shall be set to produce individual blue jets about $\frac{1}{4}$ in. in height directly over the grid without forming a common inner cone and without causing the grid to glow. With the burner placed under the center of the shield (Fig. 2), the flame shall be regulated to produce a temperature of 600 ± 15 F. at the top opening. Calibrations under the shield will afford a convenient check on the flame adjustment between successive tests. Fluctuations in gas pressure shall be determined by the use of a manometer suitably connected to the gas supply, and the pressure regulated to maintain a uniform supply to the burner.

(*c*) The burner, with calibrated flame, shall be placed directly below the center of the crib. To avoid unnecessary centering adjustments, it will be convenient to attach the legs of the ring stand permanently to the work bench and provide a receptacle or guides for the burner to insure quick and accurate placement. The top of the burner grid shall be $1\frac{1}{2}$ in. below the bottom of the lower tier of pieces in the specimen (see Fig. 3). The flame shall be applied for 3 min., after which it shall be removed by withdrawing the burner.

(*d*) The specimen shall not be disturbed until all flaming and glowing has ceased. After glowing has ceased, the specimen, including any pieces that may have fallen, shall be transferred to the scale pan of a balance and weighed.[3]

[3] Since some glow, not visible, may persist within the charcoal, the operator shall allow a reasonable time before weighing, particularly in the case of poorly treated material. For untreated wood, some charcoal may drop from the crib, and a fine-wire, ½-in. mesh screen placed on top of the ring stand, or a suitable receptacle; may be advisable to avoid loss of material.

Original and final weights shall be determined to at least the nearest 0.5 g. These weights may be determined with the specimen in the frame, deducting the weight of frame and of nails, if used.

Report

7. The original and final weights, and the moisture content of the specimen shall be recorded. The loss in weight after all flaming and glowing has ceased shall be expressed as a percentage of the original weight of the specimen as tested.[4] Duration of continued flaming and continued glow after removal of the igniting flame shall be recorded.

[4] Equipment and procedure for measurement of other properties such as rate of weight loss, maximum temperature, and time of flaming and glowing are given in the report on "Comparative Fire Tests of Treated and Untreated Wood," prepared by Subcommittee II on Fire Tests of Lumber, of Committee C-5, see *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 238 (1941).

Tentative Specifications for

85 PER CENT MAGNESIA THERMAL INSULATING CEMENT[1]

A.S.T.M. Designation: C 193 – 44 T

ISSUED, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These specifications cover 85 per cent magnesia thermal insulation in the form of dry cement or plaster, intended to be mixed with a suitable proportion of water (as recommended by the manufacturer), applied as a plastic mass, and dried in place, for use as insulation on surfaces operating at temperatures between 200 and 600 F.

Composition

2. The cement shall conform to the following requirements as to composition on the moisture-free basis:

Basic hydrated magnesium carbonate, min., per cent by weight	85
Asbestos fiber, min., per cent by weight	10

Physical Properties

3. The cement shall conform to the following requirements as to physical properties:

Bulk density, cu. ft. per 100 lb.	within plus or minus 10 per cent of manufacturer's published value
Dry covering capacity, min., bd. ft. per 100 lb. of dry cement	50
Volume change (shrinkage) upon drying, max., per cent	35
Thermal conductivity, max., Btu. in per sq. ft. per hr. per deg. Fahr.:	
At mean temperature of 200 F	0.70[a]
At mean temperature of 300 F	0.80[a]

[a] Read from the conductivity - mean temperature curve.

Sampling and Mixing

4. The cement shall be sampled and mixed, for purpose of tests, in accordance with the Standard Methods of Sampling and Mixing Thermal Insulating Cement (A.S.T.M. Designation: C 163).[3]

Methods of Testing

5. The properties enumerated in these specifications shall be determined in

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-16 on Thermal Insulating Materials.

[2] Prior to publication as tentative, these specifications were published as Emergency Specifications from 1942 to 1944.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

accordance with the following methods of the American Society for Testing Materials (except as specified in Paragraph (*a*)):

(*a*) *Composition.*—Federal Specification for Magnesia; Block, Cement, and Pipe-Covering (Molded) (HH-M-61, April 3, 1934), Sections F-3a to F-3g.

(*b*) *Bulk Density.*—Standard Method of Test for Bulk Density of Thermal Insulating Cement (A.S.T.M. Designation: C 164).[3]

(*c*) *Dry Covering Capacity.*—Tentative Methods of Test for Covering Capacity and Volume Change Upon Drying of Thermal Insulating Cement (A.S.T.M. Designation: C 166).[3]

(*d*) *Volume Change Upon Drying.*—Tentative Methods of Test for Covering Capacity and Volume Change Upon Drying of Thermal Insulating Cement (A.S.T.M. Designation: C 166).[3]

(*e*) *Thermal Conductivity.*—Tentative Method of Test for Thermal Conductivity of Materials by Means of the Guarded Hot Plate (A.S.T.M. Designation: C 177),[3] except that the test specimens shall be prepared as follows:

Test specimens shall be molded in a mold of suitable size and shape for the guarded hot plate to be used, and $1\frac{1}{2}$ in. in depth. The cement shall be mixed with water in accordance with the Standard Methods of Sampling and Mixing Thermal Insulating Cement (A.S.T.M. Designation: C 163).[3] The mold shall be placed in a horizontal position on a piece of plate glass 14 or 15 in. square. The mixed cement shall be placed in the mold, the top surface of the cement troweled smooth, and a sheet of ordinary writing paper placed on top of the cement. A steel plate 14 or 15 in. square by $\frac{1}{8}$ in. in thickness, having nine equally spaced holes $\frac{3}{16}$ in. in diameter in the central area 10 in. in diameter, shall be placed on top of the mold and a pointed pin used to puncture the paper immediately under the $\frac{3}{16}$-in. holes in the steel plate. This entire assembly shall be then inverted and the glass plate removed, the surface of the cement troweled smooth and flush with the top of the mold, and the mold then carefully removed. The perforated plate with the molded cement shall be placed on an open shelf of an oven having a volume at least 50 times that of the specimens to be dried. The test specimens shall remain in the oven for at least 48 hr. at a temperature of 225 ± 25 F. Upon removal from the oven, the surfaces of the specimens shall be made plane and parallel by cutting, sandpapering, or rubbing, or by a combination of these methods. The lateral dimensions of the specimens shall be governed by the size of the guarded hot plate apparatus.

Rejection

6. The cement may be rejected if it fails to conform to any of the requirements of these specifications. In case of dispute, a demand for a retest shall be granted.

Tentative Specifications for
ASBESTOS THERMAL INSULATING CEMENT[1]

A.S.T.M. Designation: C 194 – 44 T

ISSUED, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These specifications cover thermal insulating material composed of asbestos fiber, in the form of dry cement or plaster, which, when mixed with a suitable proportion of water (as recommended by the manufacturer), applied as a plastic mass, and dried in place, affords substantial resistance to heat transmission on surfaces operating at temperatures between 140 and 1000 F.

Composition

2. The cement shall be composed of grade 7 or longer asbestos fiber, as defined by the Quebec Asbestos Producers' Association, with a suitable proportion of heat-resistant binder.

Physical Properties

3. The cement shall conform to the following requirements as to physical properties:

Bulk density, cu. ft. per 100 lb.	within plus or minus 10 per cent of manufacturer's published value
Dry covering capacity, min., bd. ft. per 100 lb. of dry cement	18
Volume change (shrinkage) upon drying, max., per cent	40
Thermal conductivity, max., Btu. in. per sq. ft. per hr. per deg. Fahr.:	
At mean temperature of 200 F	1.30[a]
At mean temperature of 500 F	1.60[a]
At mean temperature of 700 F	1.80[a]

[a] Read from the conductivity - mean temperature curve.

Sampling and Mixing

4. The cement shall be sampled and mixed, for purpose of tests, in accordance with the Standard Methods of Sampling and Mixing Thermal Insulating Cement (A.S.T.M. Designation: C 163).[3]

Methods of Testing

5. The properties enumerated in these specifications shall be determined in accordance with the following methods

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-16 on Thermal Insulating Materials.

[2] Prior to publication as tentative, these specifications were published as Emergency Specifications from 1942 to 1944.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

of the American Society for Testing Materials:

(*a*) *Bulk Density.*—Standard Method of Test for Bulk Density of Thermal Insulating Cement (A.S.T.M. Designation: C 164).[3]

(*b*) *Dry Covering Capacity.*—Tentative Methods of Test for Covering Capacity and Volume Change Upon Drying of Thermal Insulating Cement (A.S.T.M. Designation: C 166).[3]

(*c*) *Volume Change Upon Drying.*—Tentative Methods of Test for Covering Capacity and Volume Change Upon Drying of Thermal Insulating Cement (A.S.T.M. Designation: C 166).[3]

(*d*) *Thermal Conductivity.*—Tentative Method of Test for Thermal Conductivity of Materials by Means of the Guarded Hot Plate (A.S.T.M. Designation: C 177),[3] except that the test specimens shall be prepared as follows:

Test specimens shall be molded in a mold of suitable size and shape for the guarded hot plate to be used, and $1\frac{1}{2}$ in. in depth. The cement shall be mixed with water in accordance with the Standard Methods of Sampling and Mixing Thermal Insulating Cement (A.S.T.M. Designation: C 163).[3] The mold shall be placed in a horizontal position on a piece of plate glass 14 or 15 in. square. The mixed cement shall be placed in the mold, the top surface of the cement troweled smooth, and a sheet of ordinary writing paper placed on top of the cement. A steel plate 14 or 15 in. square by $\frac{1}{8}$ in. in thickness, having nine equally spaced holes $\frac{3}{16}$ in. in diameter in the central area 10 in. in diameter, shall be placed on top of the mold and a pointed pin used to puncture the paper immediately under the $\frac{3}{16}$-in. holes in the steel plate. This entire assembly shall be then inverted and the glass plate removed, the surface of the cement troweled smooth and flush with the top of the mold, and the mold then carefully removed. The perforated plate with the molded cement shall be placed on an open shelf of an oven having a volume at least 50 times that of the specimens to be dried. The test specimens shall remain in the oven for at least 48 hr. at a temperature of 225 ± 25 F. Upon removal from the oven, the surfaces of the specimens shall be made plane and parallel by cutting, sandpapering, or rubbing, or by a combination of these methods. The lateral dimensions of the specimens shall be governed by the size of the guarded hot plate apparatus.

Rejection

6. The cement may be rejected if it fails to conform to any of the requirements of these specifications. In case of dispute, a demand for a retest shall be granted.

Tentative Specifications for
MINERAL WOOL THERMAL INSULATING CEMENT[1]

A.S.T.M. Designation: C 195 – 44 T

ISSUED, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These specifications cover thermal insulating material composed predominantly of mineral wool, in the form of dry cement or plaster, which, when mixed with a suitable proportion of water (as recommended by the manufacturer), applied as a plastic mass, and dried in place, affords substantial resistance to heat transmission on surfaces operating at temperatures between 140 and 1800 F.

Physical Properties

2. The cement shall conform to the following requirements as to physical properties:

Bulk density, cu. ft. per 100 lb	within plus or minus 10 per cent of manufacturer's published value
Dry covering capacity, min., bd. ft. per 100 lb. of dry cement	45
Volume change (shrinkage) upon drying, max., per cent	20
Thermal conductivity, max., Btu. in. per sq. ft. per hr. per deg. Fahr.:	
At mean temperature of 200 F	0.80[a]
At mean temperature of 500 F	0.90[a]
At mean temperature of 700 F	1.00[a]
Adhesion to steel, min., psi	3.0

[a] Read from the conductivity - mean temperature curve.

Sampling and Mixing

3. The cement shall be sampled and mixed, for purpose of tests, in accordance with the Standard Methods of Sampling and Mixing Thermal Insulating Cement (A.S.T.M. Designation: C 163).[3]

Methods of Testing

4. The properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(*a*) *Bulk Density.*—Standard Method of Test for Bulk Density of Thermal In-

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-16 on Thermal Insulating Materials.

[2] Prior to publication as tentative, these specifications were published as Emergency Specifications from 1942 to 1944.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

sulating Cement (A.S.T.M. Designation: C 164).[3]

(*b*) *Dry Covering Capacity.*—Tentative Methods of Test for Covering Capacity and Volume Change Upon Drying of Thermal Insulating Cement (A.S.T.M. Designation: C 166).[3]

(*c*) *Volume Change Upon Drying.*—Tentative Methods of Test for Covering Capacity and Volume Change Upon Drying of Thermal Insulating Cement (A.S.T.M. Designation: C 166).[3]

(*d*) *Thermal Conductivity.*—Tentative Method of Test for Thermal Conductivity of Materials by Means of the Guarded Hot Plate (A.S.T.M. Designation: C 177),[5] except that the test specimens shall be prepared as follows:

Test specimens shall be molded in a mold of suitable size and shape for the guarded hot plate to be used, and $1\frac{1}{2}$ in. in depth. The cement shall be mixed with water in accordance with the Standard Methods of Sampling and Mixing Thermal Insulating Cement (A.S.T.M. Designation: C 163).[3] The mold shall be placed in a horizontal position on a piece of plate glass 14 or 15 in. square. The mixed cement shall be placed in the mold, the top surface of the cement troweled smooth, and a sheet of ordinary writing paper placed on top of the cement. A steel plate 14 or 15 in. square by $\frac{1}{8}$ in. in thickness having nine equally spaced holes $\frac{3}{16}$ in. in diameter in the central area 10 in. in diameter, shall be placed on top of the mold and a pointed pin used to puncture the paper immediately under the $\frac{3}{16}$-in. holes in the steel plate. This entire assembly shall be then inverted and the glass plate removed, the surface of the cement troweled smooth and flush with the top of the mold, and the mold then carefully removed. The perforated plate with the molded cement shall be placed on an open shelf of an oven having a volume at least 50 times that of the specimens to be dried. The test specimens shall remain in the oven for at least 48 hr. at a temperature of 225 ± 25 F. Upon removal from the oven, the surfaces of the specimens shall be made plane and parallel by cutting, sandpapering, or rubbing, or by a combination of these methods. The lateral dimensions of the specimens shall be governed by the size of the guarded hot plate apparatus.

(*e*) *Adhesion to Steel.*—Adhesion of the cement to steel plate shall be determined in the following manner:

The apparatus[4] shall consist of a steel plate and a steel ring, each $10\frac{1}{4}$ in. in diameter and $\frac{3}{8}$ in. in thickness, centered and held in parallel planes 2 in. apart by four studs set in the ring. The inside diameter of the steel ring shall be $6\frac{1}{2}$ in. The material under test is held between the ring and the plate. A steel disk with mill scale intact, 6 in. in diameter and $\frac{3}{8}$ in. in thickness, shall be provided to complete the surface in the plane of the ring.

The steel ring shall be laid on a flat, horizontal plate. The steel disk shall be laid in the opening of the ring, its center concentric with that of the ring. The continuous flat surface thus presented shall be covered with a 2-in. thick troweled layer of the cement, mixed in accordance with Section 3. The ring, disk, supporting plate, and cement shall be dried to constant weight in an oven at 225 ± 25 F. The oven shall have a volume at least 50 times that of the specimens to be dried. The $10\frac{1}{4}$-in. steel plate shall be secured in place by the four studs. Hooks shall be screwed into centered holes on the outside surface of plate and disk. A measured load shall be applied to the hook in the steel

[4] The requirements for the adhesion test apparatus are identical with those specified in Section F-(2a)(5) of the Navy Department Specification for High-Temperature Insulation Cement (32C14d, November 2, 1942).

disk while the specimen is suspended from the hook in the steel plate. The load shall be increased until rupture of bond between the steel disk and the dried cement occurs. Adhesion to steel of the cement shall be expressed as pounds of load required to cause rupture of bond, per square inch of face area of the test specimen. Three determinations shall be made on each sample of cement and the average value of the three determinations shall be used.

Rejection

5. The cement may be rejected if it fails to conform to any of the requirements of these specifications. In case of dispute, a demand for a retest shall be granted.

Tentative Specifications for

EXPANDED OR EXFOLIATED VERMICULITE THERMAL INSULATING CEMENT[1]

A.S.T.M. Designation: C 196 – 44 T

ISSUED, 1944; REVISED, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These specifications cover thermal insulating material composed predominantly of expanded or exfoliated vermiculite, in the form of dry cement or plaster, intended to be mixed with a suitable proportion of water (as recommended by the manufacturer), applied as a plastic mass, and dried in place, for use as insulation on surfaces operating at temperatures between 140 and 1800 F.

Physical Properties

2. The cement shall conform to the following requirements as to physical properties:

Bulk density, cu. ft. per 100 lb.	within plus or minus 10 per cent of manufacturer's published value
Dry covering capacity, min., bd. ft. per 100 lb. of dry cement	60
Volume change (shrinkage) upon drying, max., per cent	18
Thermal conductivity, max., Btu. in. per sq. ft. per hr. per deg. Fahr.:	
At mean temperature of 200 F.	0.95[a]
At mean temperature of 500 F.	1.10[a]
At mean temperature of 700 F.	1.20[a]
Adhesion to steel, min., psi.	2.5

[a] Read from the conductivity - mean temperature curve.

Sampling and Mixing

3. The cement shall be sampled and mixed, for purpose of tests, in accordance with the Standard Methods of Sampling and Mixing Thermal Insulating Cement (A.S.T.M. Designation: C 163).[3]

Methods of Testing

4. The properties enumerated in these specifications shall be determined in accordance with the following methods

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-16 on Thermal Insulating Materials.

[2] Revision accepted by Committee E-10 on Standards, August 28, 1944.

Prior to publication as tentative, these specifications were published as Emergency Specifications from 1942 to 1944.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

of the American Society for Testing Materials:

(*a*) *Bulk Density.*—Standard Method of Test for Bulk Density of Thermal Insulating Cement (A.S.T.M. Designation: C 164).[3]

(*b*) *Dry Covering Capacity.*—Tentative Methods of Test for Covering Capacity and Volume Change Upon Drying of Thermal Insulating Cement (A.S.T.M. Designation: C 166).[3]

(*c*) *Volume Change Upon Drying.*—Tentative Methods of Test for Covering Capacity and Volume Change Upon Drying of Thermal Insulating Cement (A.S.T.M. Designation: C 166).[3]

(*d*) *Thermal Conductivity.*—Tentative Method of Test for Thermal Conductivity of Materials by Means of the Guarded Hot Plate (A.S.T.M. Designation: C 177),[3] except that the test specimens shall be prepared as follows:

Test specimens shall be molded in a mold of suitable size and shape for the guarded hot plate to be used, and 1½ in. in depth. The cement shall be mixed with water in accordance with the Standard Methods of Sampling and Mixing Thermal Insulating Cement (A.S.T.M. Designation: C 163).[3] The mold shall be placed in a horizontal position on a piece of plate glass 14 or 15 in. square. The mixed cement shall be placed in the mold, the top surface of the cement troweled smooth, and a sheet of ordinary writing paper placed on top of the cement. A steel plate 14 or 15 in. square by ⅛ in. in thickness having nine equally spaced holes $\frac{3}{16}$ in. in diameter in the central area 10 in. in diameter, shall be placed on top of the mold and a pointed pin used to puncture the paper immediately under the $\frac{3}{16}$-in. holes in the steel plate. This entire assembly shall be then inverted and the glass plate removed, the surface of the cement troweled smooth and flush with the top of the mold, and the mold then carefully removed. The perforated plate with the molded cement shall be placed on an open shelf of an oven having a volume at least 50 times that of the specimens to be dried. The test specimens shall remain in the oven for at least 48 hr. at a temperature of 225 ± 25 F. Upon removal from the oven, the surfaces of the specimens shall be made plane and parallel by cutting, sandpapering, or rubbing, or by a combination of these methods. The lateral dimensions of the specimens shall be governed by the size of the guarded hot plate apparatus.

(*e*) *Adhesion to Steel.*—Adhesion of the cement to steel plate shall be determined in the following manner:

The apparatus[4] shall consist of a steel plate and a steel ring, each 10¼ in. in diameter and ⅜ in. in thickness, centered and held in parallel planes 2 in. apart by four studs set in the ring. The inside diameter of the steel ring shall be 6¼ in. The material under test is held between the ring and the plate. A steel disk with mill scale intact, 6 in. in diameter and ⅜ in. in thickness, shall be provided to complete the surface in the plane of the ring.

The steel ring shall be laid on a flat, horizontal plate. The steel disk shall be laid in the opening of the ring, its center concentric with that of the ring. The continuous flat surface thus presented shall be covered with a 2-in. thick troweled layer of the cement, mixed in accordance with Section 3. The ring, disk, supporting plate, and cement shall be dried to constant weight in an oven at 225 ± 25 F. The oven shall have a volume at least 50 times that of the specimens to be dried. The 10¼-in. steel plate shall be secured in place by the four studs. Hooks shall be screwed into centered holes on the outside surface

[4] The requirements for the adhesion test apparatus are identical with those specified in Section F-(2a)(5) of the Navy Department Specification for High-Temperature Insulation Cement (32C14d, November 2, 1942).

of plate and disk. A measured load shall be applied to the hook in the steel disk while the specimen is suspended from the hook in the steel plate. The load shall be increased until rupture of bond between the steel disk and the dried cement occurs. Adhesion to steel of the cement shall be expressed as pounds of load required to cause rupture of bond, per square inch of face area of the test specimen. Three determinations shall be made on each sample of cement and the average value of the three determinations shall be used.

Rejection

5. The cement may be rejected if it fails to conform to any of the requirements of these specifications. In case of dispute, a demand for a retest shall be granted.

Tentative Specifications for

DIATOMACEOUS SILICA THERMAL INSULATING CEMENT[1]

A.S.T.M. Designation: C 197 – 44 T

ISSUED, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These specifications cover thermal insulating material composed predominantly of diatomaceous silica, in the form of dry cement or plaster, intended to be mixed with a suitable proportion of water (as recommended by the manufacturer), applied as a plastic mass, and dried in place, for use as insulation on surfaces operating at temperatures between 600 and 1900 F.

Physical Properties

2. The cement shall conform to the following requirements as to physical properties:

Bulk density, cu. ft. per 100 lb.	within plus or minus 10 per cent of manufacturer's published value
Dry covering capacity, min., bd. ft. per 100 lb. of dry cement	35
Volume change (shrinkage) upon drying, max., per cent	30
Thermal conductivity, max., Btu. in. per sq. ft. per hr. per deg. Fahr.:	
At mean temperature of 200 F.	0.80[a]
At mean temperature of 500 F.	1.00[a]
At mean temperature of 700 F.	1.10[a]

[a] Read from the conductivity - mean temperature curve.

Sampling and Mixing

3. The cement shall be sampled and mixed, for purpose of tests, in accordance with the Standard Methods of Sampling and Mixing Thermal Insulating Cement (A.S.T.M. Designation: C 163).[3]

Methods of Testing

4. The properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(*a*) *Bulk Density.*—Standard Method of Test for Bulk Density of Thermal Insulating Cement (A.S.T.M. Designation: C 164).[3]

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-16 on Thermal Insulating Materials.

[2] Prior to publication as tentative, these specifications were published as Emergency Specifications from 1942 to 1944.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(*b*) *Dry Covering Capacity.*—Tentative Methods of Test for Covering Capacity and Volume Change Upon Drying of Thermal Insulating Cement (A.S.T.M. Designation: C 166).[3]

(*c*) *Volume Change Upon Drying.*—Tentative Methods of Test for Covering Capacity and Volume Change Upon Drying of Thermal Insulating Cement (A.S.T.M. Designation: C 166).[3]

(*d*) *Thermal Conductivity.*—Tentative Method of Test for Thermal Conductivity of Materials by Means of the Guarded Hot Plate (A.S.T.M. Designation: C 177),[3] except that the test specimens shall be prepared as follows:

Test specimens shall be molded in a mold of suitable size and shape for the guarded hot plate to be used, and 1½ in. in depth. The cement shall be mixed with water in accordance with the Standard Methods of Sampling and Mixing Thermal Insulating Cement (A.S.T.M. Designation: C 163).[3] The mold shall be placed in a horizontal position on a piece of plate glass 14 or 15 in. square. The mixed cement shall be placed in the mold, the top surface of the cement troweled smooth, and a sheet of ordinary writing paper placed on top of the cement. A steel plate 14 or 15 in. square by ⅛ in. in thickness, having nine equally spaced holes $\frac{3}{16}$ in. in diameter in the central area 10 in. in diameter, shall be placed on top of the mold and a pointed pin used to puncture the paper immediately under the $\frac{3}{16}$-in. holes in the steel plate. This entire assembly shall be then inverted and the glass plate removed, the surface of the cement troweled smooth and flush with the top of the mold, and the mold then carefully removed. The perforated plate with the molded cement shall be placed on an open shelf of an oven having a volume at least 50 times that of the specimens to be dried. The test specimens shall remain in the oven for at least 48 hr. at a temperature of 225 ± 25 F. Upon removal from the oven, the surfaces of the specimens shall be made plane and parallel by cutting, sandpapering, or rubbing, or by a combination of these methods. The lateral dimensions of the specimens shall be governed by the size of the guarded hot plate apparatus.

Rejection

5. The cement may be rejected if it fails to conform to any of the requirements of these specifications. In case of dispute, a demand for a retest shall be granted.

Tentative Methods of Test for

COVERING CAPACITY AND VOLUME CHANGE UPON DRYING OF THERMAL INSULATING CEMENT[1]

A.S.T.M. Designation: C 166 – 41 T

ISSUED, 1941.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. Wet covering capacity and volume change upon drying are often of major importance in the application of thermal insulating cement. These properties can be easily determined at the same time that the determinations of dry covering capacity are made. Therefore, the procedures for determining these three properties are covered together in these methods.

Definitions

2. (*a*) *Wet Covering Capacity.*—The wet covering capacity is the volume occupied in "board feet per 100 lb. of dry cement" when the cement is mixed with the recommended amount of water and molded.

(*b*) *Dry Covering Capacity.*—The dry covering capacity is the volume occupied in "board feet per 100 lb. of dry cement" after the wet cement is molded and dried to constant weight in accordance with Section 5.

(*c*) *Volume Change upon Drying.*—The volume change upon drying is the percentage change in volume of the molded cement mixed with the recommended amount of water when dried to constant weight in accordance with Section 5.

Apparatus

3. The apparatus shall consist of the following:

(*a*) *Mold.*—A rigid mold having inside dimensions of 1 by 12 by 36 in. with one end and one face open, and a piece of wood or other suitable material 1 by 2 by 11 $\frac{7}{8}$ in. in dimensions for squaring up the end of the test specimen toward the open end of the mold.

(*b*) *Wax Paper.*—Sheets of wax paper 12 by 36 in. in dimensions.

(*c*) *Engine Oil.*

(*d*) *Trowel.*—A 16-in. rectangular plasterer's trowel.

(*e*) *Steel Rules.*—Steel rules 18 and 36 in. in length, accurate to within $\frac{1}{64}$ in.

(*f*) *Depth Gage.*—A depth gage consisting of a rigid, pointed rod approximately $\frac{1}{8}$ in. in diameter fitted with a

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee C-16 on Thermal Insulating Materials.

[2] Accepted by the Society at annual meeting, June, 1941.

flat disk about ½ in. in diameter which may be moved along the rod either by a sliding action or by means of threads.

Sampling and Mixing

4. The cement shall be sampled and mixed in accordance with the Standard Methods of Sampling and Mixing Thermal Insulating Cement (A.S.T.M. Designation: C 163) of the American Society for Testing Materials.[3]

Procedure

5. (*a*) The inside surfaces of the mold shall be well oiled or lined with wax paper in order to prevent the cement from sticking to the sides and to permit convenient removal of the specimen. The mixed cement shall then be placed in the mold.

(*b*) The cement shall be troweled in one layer and in two directions lengthwise of the mold without unnecessary compacting, until the surface is smooth and flush with the top edges of the mold. Sufficient wet cement shall be used to fill the mold when the piece of wood or other suitable material is in place in the open end of the mold and in contact with the cement in order to make a square end and to prevent movement of the cement after the troweling is completed.

(*c*) The thickness shall be measured at nine points on the top surface of the sample as indicated in Fig. 1.

(*d*) Thickness measurements shall be made within an accuracy of plus or minus 2 per cent by means of a depth gage. The pointed rod of the depth gage shall be carefully pushed through the cement until it comes in contact with the bottom of the mold. The rod shall be constantly held in a vertical position and the disk shall be adjusted until its flat surface just comes into contact with the top surface of the cement. The disk shall be secured to the pin before the gage is withdrawn from the cement. The thickness of the cement shall be determined by measuring the distance from the flat surface of the disk to the pointed end of the rod with a steel rule or by some other suitable scale. The indentations in the cement showing the points of measurement shall not be filled but shall be allowed to dry in place.

(*e*) The width and length measurements shall be made with the steel rules within an accuracy of plus or minus $\frac{1}{16}$ in. Width measurements shall be made at

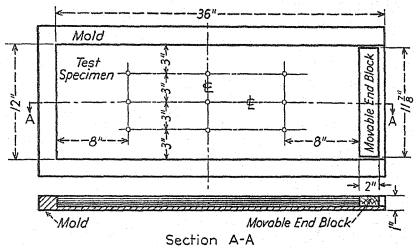


FIG. 1.—Mold for Test Specimens and Locations of Points for Thickness Measurements.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

locations approximately 6 in. from each end of the specimen. Length measurements shall be made at locations approximately 2 in. from each side of the specimen. While making these measurements, the edge of the steel rule shall be allowed to make an indentation in the wet cement in order to assure measurements at the same locations after the cement has been dried.

(*f*) Immediately after the measurements have been made, or as soon thereafter as recommended by the manufacturer, the cement specimen and mold shall be placed in an oven at 225 ± 25 F. until dried to constant weight. The oven shall be adequately vented and shall have a volume at least 50 times that of the specimens in the oven.

(*g*) After the dried specimen is weighed, it shall be measured for thickness, width, and length at the same locations at which corresponding measurements were made before the specimen was dried. These locations are indicated by the indentation marks made in the wet cement. The thickness shall be measured as described in Paragraph (*d*). In order to prevent false thickness measurements on the dry specimen, due to the bottom surface of the specimen convexing, the specimen shall be removed from the mold and a flexible steel rule placed so as to conform to the bottom surface of the specimen at points where the thickness is to be measured. The point of the depth gage shall be inserted from the original top face until it touches the steel rule.

Calculations

6. The wet and dry covering capacities and the volume change upon drying shall be calculated as follows:

$$C_w = \frac{dbl}{144W} \times 100$$

$$C_d = \frac{d_1 b_1 l_1}{144W} \times 100$$

$$V = \frac{(dbl) - (d_1 b_1 l_1)}{dbl} \times 100$$

where:

C_w = wet covering capacity in board feet per 100 lb. of dry cement,

C_d = dry covering capacity in board feet per 100 lb. of dry cement,

V = percentage volume change upon drying,

d = average thickness of wet specimen in inches,

b = average width of wet specimen in inches,

l = average length of wet specimen in inches,

W = weight of dry specimen in pounds,

d_1 = average thickness of dry specimen in inches,

b_1 = average width of dry specimen in inches, and

l_1 = average length of dry specimen in inches.

NOTE.—Due to the wax paper or oil treatment preventing adhesion between cement and mold, the volume change (shrinkage) on drying will usually be greater in this test than that encountered in service where adhesion is obtained between the cement and the surface on which it is applied.

Tentative Methods of Test for

COMPRESSIVE STRENGTH AND FLEXURAL STRENGTH OF PREFORMED BLOCK TYPE THERMAL INSULATING MATERIALS[1]

A.S.T.M. Designation: C 165 - 41 T

ISSUED, 1941.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These methods of test cover procedures for determining the compressive strength and flexural strength of preformed block type thermal insulating materials.

NOTE.—These methods are not applicable to certain types of thermal insulating materials.

COMPRESSIVE STRENGTH

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Testing Machine.*—Any form of standard hydraulic or mechanical compression testing machine.

(*b*) *Spherical Bearing Block.*—A spherical bearing block having a plane bearing surface at least 6 in. square.

Test Specimens

3. (*a*) The test specimens shall be preferably 6 in., but in no case less than 5 in., square.

NOTE.—When comparative tests are to be made on preformed materials, all test specimens shall be of the same thickness, preferably 1½ in.

(*b*) The specimens shall be cut from larger blocks or irregular shapes in such a manner as to preserve as many of the original surfaces as possible. Only one specimen shall be cut from a single block or shape. The bearing faces of the test specimens shall be approximately parallel planes. Where the original surfaces of the block are substantially plane and parallel, no special preparation of the surfaces will usually be necessary. In preparing specimens from pieces of irregular shape, any means such as a band saw, or any method involving the use of abrasives such as a high-speed abrasion wheel or a rubbing bed, that will produce a specimen with approximately plane and parallel faces without weakening the structure of the specimen may be used.

(*c*) The test specimens shall be dried in a vented oven for not less than 16 hr. at 225 ± 25 F.

Procedure

4. (*a*) At least four specimens shall be tested. In the case of blocks 3 in. in

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committeee C-16 on Thermal Insulating Materials.

[2] Accepted by the Society at annual meeting, June, 1941.

width, two pieces each 6 in. in length shall be cut from the same block and tested side by side simultaneously; the two pieces shall count as one test specimen.

(*b*) The specimens shall be tested immediately upon removal from the drying oven.

(*c*) The load shall be applied perpendicular to the square face of the test specimen.

(*d*) The bearing block shall be used on top of the test specimen in vertical testing machines when only one bearing block is provided. The plane bearing surface of the block assembly shall be in

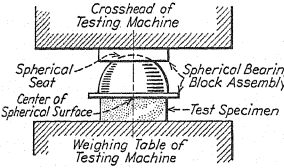


FIG. 1.—Spherical Bearing Block for Compressive Strength Test.

contact with the entire area of the top square face of the test specimen, and the bearing surface shall be parallel to the weighing table of the testing machine (Fig. 1). The spherical seat of the bearing block shall be kept thoroughly lubricated to insure accurate adjustment, which should be made by hand under an initial load of 2 psi.

(*e*) The speed of the moving head of the testing machine shall be not more than 0.05 in. per min.

(*f*) The specimen shall be compressed to a deformation of 5 per cent of its original thickness unless definite failure shall have occurred before this deformation is reached. The loads required to produce failure and/or deformations of 1, 2, 3, 4, and 5 per cent shall be recorded.

Calculations

5. (*a*) The compressive strength shall be calculated as follows:

$$S = \frac{W}{A}$$

where:

S = compressive strength in pounds per square inch,

W = load in pounds at 5 per cent deformation or at failure, whichever is smaller, and

A = average of the gross areas of the top and bottom faces of the specimen in square inches.

(*b*) The stress in pounds per square inch required to produce deformations of 1, 2, 3, and 4 per cent shall also be calculated from the formula given in Paragraph (*a*).

Report

6. The report shall include the following:

(*1*) The average value of the compressive strength in pounds per square inch, and

(*2*) The average values of the stresses in pounds per square inch required to produce deformations of 1, 2, 3, and 4 per cent.

FLEXURAL STRENGTH

Apparatus

7. The apparatus shall consist of the following:

(*a*) *Testing Machine.*—Any form of standard testing machine capable of applying and measuring the required load within an accuracy of plus or minus 2 per cent.

(*b*) *Bearing Edges.*—Cylindrical bearing edges $1\frac{1}{4} \pm \frac{1}{4}$ in. in diameter. The bearing cylinders shall be straight and shall be self-aligning so as to maintain full contact with the specimen throughout the test. They shall have a length at least equal to the width of the specimen.

NOTE.—The apparatus shown in Fig. 2 of the Standard Methods of Test for Cold Crushing Strength and Modulus of Rupture of Refractory Brick and Shapes (A.S.T.M. Designation: C 133) of the American Society for Testing Materials[3] is an acceptable form but of shorter span than is specified in Section 9 (*b*) of these methods.

Test Specimens

8. (*a*) The test specimens shall be preferably 6 in., but in no case less than 3 in., in width and shall be approximately 12 in. in length.

NOTE.—If the test specimen is cut to obtain a narrower width than as-received, the cut shall be made lengthwise of the block. When comparative tests are to be made on preformed materials, all specimens shall be of the same thickness, preferably $1\frac{1}{2}$ in.

(*b*) Test specimens shall be prepared in accordance with Section 3 (*b*) and (*c*).

Procedure

9. (*a*) At least four specimens shall be tested.

(*b*) The bearing edges shall be placed so as to provide a span of 10 in. A test specimen shall be placed flatwise on the bearing edges and the load applied at mid-span, across the width and perpendicular to the top face of the specimen.

(*c*) The speed of the moving head of the testing machine, during the application of the load, shall be not more than 0.05 in. per min., or the rate of loading shall be not more than 500 lb. per min.

(*d*) The load shall be applied until definite failure occurs, and the maximum load in pounds as indicated by the testing equipment shall be recorded.

Calculation

10. The flexural strength shall be calculated as follows:

$$R = \frac{3Wl}{2bd^2}$$

where:

R = flexural strength in pounds per square inch,
W = load in pounds at which the specimen failed,
l = distance between the supports in inches,
b = width of the specimen in inches, and
d = thickness of the specimen in inches.

Report

11. The average value of the flexural strength in pounds per square inch shall be reported.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Tentative Method of Test for

THERMAL CONDUCTIVITY OF MATERIALS BY MEANS OF THE GUARDED HOT PLATE[1]

A.S.T.M. Designation: C 177 – 42 T

ISSUED, 1942.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

This method was developed by the Joint Committee on Thermal Conductivity of All Forms of Insulation, sponsored by the A.S.H.V.E., A.S.T.M., A.S.R.E., and N.R.C.

Introduction

1. (*a*) This method describes procedures to be used in the determination, by means of the guarded hot plate, of the thermal conductivity of insulating, building, and other materials, whose conductivities do not exceed the maximum value for which these procedures are applicable as specified in Section 2.

(*b*) Because of the requirements prescribed in this method as to conditions under which conductivity tests shall be made, it should be recognized that the conductivity coefficients obtained will not necessarily be the values pertaining under all service conditions. As an example, the method provides that the conductivity coefficients shall be obtained by test on dry specimens, while in service such a condition will seldom be realized.

(*c*) The guarded hot plate is generally used for determining the thermal conductivity of homogeneous materials in the form of flat slabs, and this method covers the procedure for such tests. It is recognized, however, that it is frequently desirable to determine the conductivity of certain materials used as pipe coverings, etc., and also materials constituting a wall construction or a part thereof. For such purposes the guarded-end or calibrated-end pipe methods of test and the guarded hot box method are recommended.

(*d*) For satisfactory results, the principles governing the size, construction, and use of apparatus for the test described in this method should be followed. If the results are to be reported as having been obtained by this method, then all of the requirements prescribed in this method shall be met.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-16 on Thermal Insulating Materials.

[2] Accepted by the Society at annual meeting, June, 1942.

(*e*) It should be recognized that it is impossible in a method of this type to establish details of construction and procedure covering all contingencies so that it can be followed by a nontechnical person; and that, on this account, technical knowledge on the part of those using this method concerning the theory of heat flow, temperature measurement, and general testing practices cannot be dispensed with as a result of the standardization of the method. It is further recognized that it would be unwise, because of the standardization of this method, to restrict in any way the activities of research workers in the further development of new and improved methods.

Scope

2. (*a*) For practical purposes, this method of test is limited to the determination of the thermal conductivity of materials having conductivities not in excess of 5.0 Btu. in. per sq. ft. per hr. per deg. Fahr.

(*b*) The method is further limited in its application to thermal conductivity tests between the extreme temperatures of −50 F. and 1400 F., or between mean temperatures of approximately 0 F. and 1200 F.

Symbols and Definitions

3. (*a*) *Symbols.*—The symbols used in this method have the following significance:

Q = total quantity of heat transferred,

τ = time,

$q = \frac{Q}{\tau}$ = rate of heat flow,

A = actual area normal to the path of heat flow (flat surface),

k = thermal conductivity,

t_1 = temperature of the hot surface,

t_2 = temperature of the cold surface,

L = length of path of heat flow (thickness),

R = thermal resistance (degrees, per unit of heat transferred per unit time), and

$C = \frac{1}{R}$ = thermal conductance (heat transferred per unit time, per degree).

(*b*) *Thermal Conductivity.*—The thermal conductivity of a homogeneous material is the rate of heat flow, under steady conditions, through unit area, per unit temperature gradient in the direction perpendicular to the area. It is calculated as follows:

$$k = \frac{QL}{A\tau(t_1 - t_2)} = \frac{qL}{A(t_1 - t_2)} \quad \ldots\ldots (1)$$

NOTE 1.—Thermal conductivity is usually expressed in English units as Btu. per sq. ft. per hr. per deg. Fahr. for a thickness of 1 in. This may be expressed mathematically as Btu. in. per hr. sq. ft. deg. Fahr., or Btu. hr.$^{-1}$ ft.$^{-2}$ deg. Fahr.$^{-1}$ in., or in various other forms. Other units are also found in the literature, and the conversion factors given in Table I may therefore be found useful.

TABLE I.—CONVERSION FACTORS FOR THERMAL CONDUCTIVITY.

	Cal. cm. per sec. per sq. cm. per deg. Cent.	Watts cm. per sq. cm. per deg. Cent.	Kg-cal. m. per hr. per sq.m. per deg. Cent.	Btu. in. per sq. ft. per hr. per deg. Fahr.
1 cal. cm. per sec. per sq. cm. per deg. Cent.	1	4.185	360	2903
1 watt cm. per sq. cm. per deg. Cent.	0.2389	1	86.00	693.7
1 kg-cal. m. per hr. per sq. m. per deg. Cent.	0.002778	0.01163	1	8.064
1 Btu. in. per sq. ft. per hr. per deg. Fahr.	0.0003445	0.001442	0.1240	1

(*c*) *Thermal Conductance.*—The thermal conductance of a body between two

specified isothermal surfaces is equal to the rate of heat flow through the body, per unit difference between the temperatures of the isothermal surfaces under consideration. It is calculated as follows:

$$C = \frac{1}{R} = \frac{q}{(t_1 - t_2)} \quad \ldots\ldots\ldots\ldots (2)$$

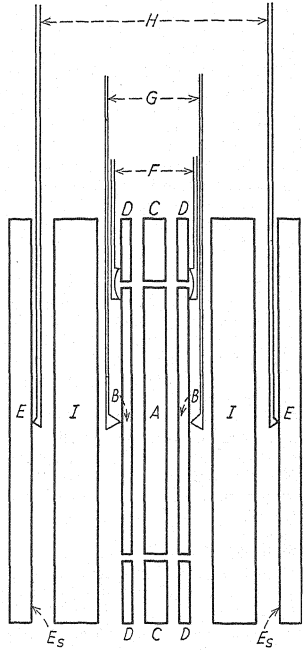
NOTE 2.—Thermal conductance is usually expressed in English units as Btu. per hr. per deg. Fahr. temperature difference between the two surfaces. Recent developments in nomenclature have largely eliminated the term C in favor of the expression $1/R$. The heating and ventilating engineer, however, dealing largely with compound walls, makes considerable use of the term unit conductance or C/unit area, and has, therefore, continued the use of the term C for conductance.

Apparatus

4. (*a*) It is not intended in this method to include detailed requirements for the construction or operation of any particular guarded hot plate for determining conductivity values. Any plate conforming to the limitations prescribed in Paragraphs (*b*) to (*i*) will be satisfactory.

NOTE.—For the convenience of new workers in the field, arrangements have been made for making available complete specifications and directions for the building and use of three typical guarded hot plates[2] complying with the requirements of this method and known as the National Bureau of Standards plate, the National Research Council plate, and the Alundum plate. The National Bureau of Standards plate and the National Research Council plate are square and have metal surface plates. The Alundum plate is built with the heating coils molded into alundum cement so as to give a circular plate with alundum cement faces. It has the obvious advantage of being usable in the moderate high temperature field.

(*b*) The general features of the guarded hot plate are shown in Fig. 1. The plates are usually square, but round plates are occasionally used. The term guarded hot plate is applied to the entire assembled apparatus including the



Heating Unit { A—Central Heater. B—Central Surface Plates. } Central Section of Heating Unit.
{ C—Guard Heater. D—Guard Surface Plates. } Guard Section of Heating Unit.

E—Cooling Units.
E_S—Cooling Unit Surface Plates.
F—Differential Thermocouples.
G—Heating Unit Surface Thermocouples.
H—Cooling Unit Surface Thermocouples.
I—Test Specimens.

FIG. 1.—General Features of the Guarded Hot Plate.

[2] Detailed information on each of the three guarded hot plates mentioned is available from the American Society for Testing Materials, 260 S. Broad St., Philadelphia 2, Pa.

heating unit and the cooling units. The heating unit consists of the central section of the heating unit and the guard section of the heating unit. The central section of the heating unit consists of a central heater and central surface plates. The guard section of the heating unit consists of one or more guard heaters and the guard surface plates. The surface plates are usually made of noncorrosive, highly conducting metal; however, the central section and the guard section of the heating unit may each be molded of alundum cement or other materials of low conductivity so as to include both the heater and the surface plates.

(*c*) In the design of the guarded hot plate for testing materials for use in any particular temperature or conductivity range, due consideration shall be given to the materials used in the construction of the hot plate with respect to their performance at the temperature to which the plate will be subjected. Consideration shall also be given to the rate at which heat must be supplied and absorbed by the heater and the coolers in designing the electrical resistance and current-carrying capacity of the heating element. Otherwise, no particular variations need be made in the design of hot plates for use in determining the conductivity of materials at the widely different mean temperatures mentioned in Section 2 (*b*).

(*d*) Heating units having metallic surfaces shall have a definite separation or air gap not greater than $\frac{1}{8}$ in. between the measuring area of the central surface plate and the guard surface plates. These plates shall have highly emissive surfaces. The separation between the main central section and the guard section of the heating units shall not exceed $\frac{3}{4}$ in., and this separation is allowable only if the spacing bars on either side of the plate separation are of heavy copper in order to distribute the heat on the surface plates. In all other cases the separation shall not exceed $\frac{1}{8}$ in. The test area shall be calculated from the center of one separation to the center of the other separation across the central surface of the plate.[4]

(*e*) The heater of the guarded hot plate shall be provided with at least two thermocouples on each face of the central surface plate, and at least two thermocouples on each face of the guard surface plate, located at opposite edges. These thermocouples may be read either individually to indicate any temperature difference that may exist between the central and guard surface plates or they may be connected differentially, and thus indicate such temperature difference directly. The differential method of connecting the center-to-guard thermocouples is the more sensitive, and is to be preferred. When the thermocouples are to be read individually, they may be peened, welded, or soldered to the surface plates. When they are to be connected differentially, it is essential that they be electrically insulated from each other. This may be accomplished by installing the thermocouples in shallow grooves in the surface plates with an electrically insulating cement, in such a manner that the bead of each thermocouple is in the plane of the surface of the plate.

(*f*) The cooling units shall have the same surface dimensions as the heating unit. They may consist of metallic plates cooled by a fluid, electrically heated plates maintained at temperatures below that of the heating unit, or thermal insulation applied to the cool

[4] In putting a new heating unit into operation for the first time, care should be taken to insure that the two faces of the heating unit maintain essentially equal temperature throughout the temperature range of the apparatus.

surface of the test specimen, depending on the mean temperature desired.

(*g*) The surface temperatures of the test specimen may be determined either by means of thermocouples mounted in the hot and cold plates or by thermocouples located in the surface of the specimen, depending on the nature of the material to be tested. For nonrigid materials that have a unit conductance of less than 1.0 Btu. per sq. ft. per hr. per deg. Fahr. and that conform to the surfaces of the plates, the surface temperatures of the specimens shall be taken as those indicated by the thermocouples attached to the hot and cold surface plates. For this purpose both the hot and cold plates shall each be supplied with at least two thermocouples mounted in the surfaces of the plates as described in Paragraph (*e*). Preferably, the thermocouples should be insulated from the plates and read differentially between the hot and cold plates, the surface temperatures of the specimen being taken as those of the plate surfaces in contact with it. For rigid materials that fail to conform to the surfaces of the plate, and for all materials having a unit conductance higher than 1.0 Btu. per sq. ft. per hr. per deg. Fahr., separate surface thermocouples shall be used. These thermocouples shall be mounted on the surface of the specimen in any convenient manner suitable for the purpose, such that the thermocouple junction is flush with the surface of the specimen. When thermocouples are used in the surface of the specimen, there shall be placed between the surfaces of the specimen and the hot and cold plates a piece of blotting paper or asbestos paper, depending on the temperatures encountered. For nonrigid materials of low conductivity, the use of thermocouples in the plates is preferred; while for rigid materials and those of high conductivity, the use of surface thermocouples is preferred. In the intermediate range the choice of method is left to the judgment of the operator.

(*h*) The thermocouples mounted in the surfaces of the plates shall be made of wire not larger than No. 23 A.w.g., while those used as surface thermocouples shall be made of wire not larger than No. 29 A.w.g.

(*i*) A potentiometer having a sensitivity of 5 microvolts or less shall be used for all measurements of electromotive force.

Sampling and Preparation of Specimens

5. (*a*) When this method is used as a guide in determining the thermal conductivity of special samples for control of manufacturing processes, for determining compliance with purchase specifications, and for other similar purposes, the selection and preparation of samples must obviously be left to the discretion of the person desiring the information. When, however, standard tests are to be made for the purpose of reporting thermal conductivity of a given material for consumer use, or for any other purpose where a definite statement of the history and condition of the sample is not available, and in all cases where the test is reported as having been performed in accordance with this method, the material shall be sampled and the specimens shall be prepared in accordance with Paragraphs (*b*) to (*h*).

(*b*) The sample shall be selected so as to provide two specimens as nearly identical as possible and of such size as to completely cover the heating unit. The specimens shall be of sufficient thickness to give a true average representation of the insulating material to be tested. Since excessive thickness of the speci-

mens and consequent excessive dimensions of the area of the guarded hot plate unnecessarily complicate the manipulation of the test procedure, the specimens shall not be too thick. The thickness of the specimen shall be great enough to allow a sufficiently accurate measurement of this dimension for calculating the thermal conductivity. The relationship between the maximum thickness of the test specimen used and the minimum dimensions of the guarded hot plate shall be as follows:

Maximum Thickness of Test Specimen, in.	Minimum Linear Surface Dimensions of Guarded Hot Plate (Square or Round), in.	
	Central Section or Test Area of Heating Unit	Guard Section or Guard Area of Heating Unit
1	4	1½
1½	8	2¾
2	12	3
4	12	6

(*c*) The sample shall be chosen as a fair representative of the material of the particular type on the market, or that to be used by the consumer. It should, therefore, preferably be purchased on the open market by an unbiased person. Three separate samples, not expected to have originated from the same day's output at the manufacturing plant, nor from a single shipment therefrom, shall be thus obtained from three different sources. The three or four tests prescribed in Section 6 (*a*) shall be made on each of these three samples in determining the conductivity of the given material.

(*d*) In testing all forms of homogeneous materials, the surfaces of the test specimens shall be made as plane as possible, by sandpapering or otherwise, in order that intimate contact between the specimens and the plates or the paper may be effected.

(*e*) The sample from which the test specimens are to be taken shall be weighed in the as-received condition and then dried at 215 F. until excess moisture is driven off as indicated by a constant-weight determination. (If the material is one that may be chemically affected by heating to 215 F., the sample shall be dried in a desiccator at from 120 to 140 F.) The as-received weight, the dry weight, and the necessary physical dimensions of the sample for calculation of the density of the material as tested shall be recorded. (For solid and blanket-type materials the physical dimensions shall be determined separately before and after drying. The density of loose-fill materials shall be based on the volume occupied in the guarded hot plate and the weight after drying.)

(*f*) Samples of homogeneous solid materials to be used for test shall be dried in accordance with Paragraph (*e*), and the specimens cut to size, or molded or pressed into the proper size and shape, with due consideration to the treatment of the material in question, weighed, and placed in the guarded hot plate for measurement of thickness, prior to testing, in accordance with Section 6 (*c*).

(*g*) Samples of blanket-type materials to be used for test shall, after being dried in accordance with Paragraph (*e*), be measured for thickness in accordance with the Standard Methods of Test for Thickness and Density of Blanket Type Thermal Insulating Materials (A.S.T.M. Designation: C 167) of the American Society for Testing Materials.[5] Two representative specimens of the material cut to proper size shall be weighed, then placed in the guarded hot plate and compressed to the specified thickness, and the thickness measured in accordance with Section 6 (*c*).

(*h*) Samples of loose-fill materials to be tested at any prescribed density

[5] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

shall, after drying in accordance with Paragraph (*e*), be confined in the shape of a square or circular slab of dimensions suitable for the particular hot plate to be used and of a thickness not less than five times the size of any particle composing the test specimen, provided that in no case shall the thickness be less than 1 in. Two representative portions of the sample, of such weight (determined to an accuracy of plus or minus 0.5 per cent) as will give the prescribed density when packed into spaces of the required dimensions, shall be weighed out and fluffed up. The test specimens shall then be prepared in accordance with either method 1 or 2, as follows:

Method 1.—The guarded hot plate shall be set up with the required distances between the heating unit and the cold plates. Each portion of the sample shall be divided into four equal parts. One part shall be placed in one side of the apparatus and vibrated or tamped until it occupies just one-quarter of the volume of that side. The remaining parts shall be introduced in the same way, one at a time, packing the material down by vibrating or tamping until it occupies its appropriate volume. The other sample shall then be placed in the other side of the apparatus in exactly the same manner.

Method 2.—Two shallow square or circular boxes having outside flat dimensions the same as those of the guarded hot plate shall be used. The edges shall be made of wood strips $\frac{1}{4}$ in. in thickness and of such width as to make the depth of the box equal to the thickness of the specimen to be tested. The two square or circular faces shall be made by gluing blotting paper or asbestos paper to the edges of the strips. With one face in place and the boxes lying horizontally, one portion of the sample shall be placed in each box, the material pressed down, and the other paper face glued in place. These two boxes containing the test specimens shall then be placed in the guarded hot plate. For materials the densities of which cannot be altered at will, the container shall be placed in a horizontal position with one side open, an excess of the dried sample weighed and poured in, the container shaken, and the excess material leveled before the upper paper face is glued in place. The weight of the material used shall be obtained by weighing the excess and subtracting it from the weight of the original material.

Procedure

6. (*a*) At least three and preferably four determinations shall be made on each specimen at mean temperatures which will cover the range of temperatures over which the material is to be used. For any test, the temperature difference across the specimen shall be not less than 40 F. The above mean temperatures of the specimen shall differ from each other by at least 30 F.

(*b*) The atmosphere surrounding the test equipment shall have a dew-point temperature not higher than the coolest part of any surface or material in, or forming a part of, the test apparatus.

(*c*) If the thermocouples mounted in the surfaces of the plates are used to determine surface temperatures (Section 4 (*g*)) the thickness of the test specimen shall be taken as the distance between the surfaces of the hot and cold plates when the specimen is in place in the apparatus. If separate surface thermocouples are used, the thickness of the test specimen shall be taken as the distance between the surfaces of the hot and cold plates when the specimen is in place in the apparatus, minus the thickness of the two layers of blotting or asbestos paper used.

(*d*) The heating element of the central heater shall be supplied with electrical energy regulated to give the desired temperature gradient through the test specimen and held constant within plus or minus 1 per cent, means being provided to measure this energy. Automatic regulation is recommended. Where automatic regulation is not available, the energy input shall be regulated by means of manual adjustment. The rate of electrical energy input to the guard ring shall then be adjusted so that the maximum temperature difference (Paragraph (*e*)) between the center and guard surface plates during the 5-hr. test observation period shall be not greater than 0.75 per cent of the average temperature drop through the two halves of the specimen, as determined by the differential thermocouples or the surface thermocouples. The average temperature difference between these surface plates during the test period shall be not greater than 0.2 per cent of the temperature drop through the specimen.

(*e*) The cooling units shall be so adjusted that the temperature drops through the two test specimens shall not differ by more than 1 per cent.

(*f*) After steady state has been reached, the test shall be continued with the necessary observations being made to determine temperature difference, center-to-guard thermal balance, and heat input until successive observations made at intervals of not greater than 1 hr., over a period of 5 hr., give thermal conductivity values that are constant to within 1 per cent.

(*g*) Upon completion of the test, the specimen shall be reweighed and the weight recorded.

Calculations

7. (*a*) The density of the sample after drying, the moisture in the sample as received, and the moisture regain during test shall be calculated as follows:

$$D = \frac{A}{B}$$

$$M = \frac{C - A}{B}$$

$$R = (G - F) \times \frac{A}{F} \times \frac{1}{B}$$

where:

D = density of the sample after drying (pounds per cubic inch or grams per cubic centimeter),

M = moisture in the sample as received (pounds per cubic inch or grams per cubic centimeter),

R = moisture regain during test (pounds per cubic inch or grams per cubic centimeter),

A = weight of sample after drying prior to test (pounds or grams),

B = volume of sample after drying (cubic inches or cubic centimeters),

C = weight of sample as received (pounds or grams),

F = weight of specimen prior to test (pounds or grams), and

G = weight of specimen after test (pounds or grams).

(*b*) Thermal conductivity shall be calculated by means of Eq. 1 (Section 3 (*b*)).

Report

8. (*a*) The report of the results of each test shall include the following:

(*1*) Name and any other identification of the material,

(*2*) Thickness of specimen tested,

(*3*) Dry weight of sample before test,

(*4*) Density of dried sample (before test),

(*5*) Moisture in sample "as received,"

(*6*) Moisture regain during test,

(*7*) Temperature range of test,

(*8*) Hot-surface temperature,

(*9*) Cold-surface temperature,

(*10*) Mean temperature of test,

(*11*) Heat input in Btu. per sq. ft. per hr., and

(*12*) Thermal conductivity.

(*b*) The conductivity - mean temperature relationship for any material tested shall be obtained from a curve resulting from plotting the thermal conductivity *versus* mean temperature, and representing the average of the tests on the three samples tested. The maximum deviation obtained from this average curve, and the mean temperature at which this maximum occurs shall be reported.

Tentative Specifications for

AIR-SETTING REFRACTORY MORTARS (WET TYPE) FOR BOILER AND INCINERATOR SERVICES[1]

A.S.T.M. Designation: C 178 - 44 T

ISSUED, 1943; REVISED, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These specifications cover the wet type of air-setting refractory mortars for use in laying up fireclay refractories in boiler furnaces and incinerators. Requirements are specified for three classes of mortars. The mortar used should be of sufficient refractoriness for the type of service encountered. Unless otherwise specified, high heat duty mortar shall be supplied.

Classes

2. High-temperature refractory mortars are classified according to the service and grade of fireclay refractories with which they are to be used. The several classes of mortars are distinguished by their refractoriness test temperatures (Section 4 (*d*)), as follows:

Class	Refractoriness Test Temperature
Super duty	2910 F. (1600 C.)
High heat duty	2730 F. (1500 C.)
Intermediate heat duty	2550 F. (1400 C.)

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-8 on Refractories.

[2] Revision accepted by the Society at annual meeting, June, 1944.

Quality

3. The mortar shall be sufficiently free from grit and of such quality and plasticity that it will spread satisfactorily with a trowel, either as it comes from the container or after a moderate amount of tempering with water. The mortar shall have properties that will enable it to be converted readily to dipping consistency by the addition of water. At any time within a 6-month period after purchase, the mortar in a newly opened container shall not have stiffened or hardened to such an extent as to prevent its easy removal and mixing.

Test Requirements

4. (*a*) *Particle Size.*—The particle size of the mortar shall be such that 95 per cent shall pass a No. 40 (420-micron) A.S.T.M. sieve (equivalent to mesh No. 35 of the Tyler standard series), and not more than 0.5 per cent shall be retained on a No. 20 (840-micron) A.S.T.M. sieve (equivalent to mesh No. 20 of the Tyler standard series).

(*b*) *Water Content.*—The total water content of the mortar shall be not more than 25 per cent, calculated on the wet basis.

(*c*) *Bonding Strength.*—The mortar when made into a joint shall have a modulus of rupture of not less than 200 psi.

(*d*) *Refractoriness.*—The mortar shall not flow out of the joint when it is subjected to the refractoriness test at the following temperature:

Class	Refractoriness Test Temperature
Super duty	2910 F. (1600 C.)
High heat duty	2730 F. (1500 C.)
Intermediate heat duty	2550 F. (1400 C.)

Retests

5. Because of variables resulting from sampling and the lack of satisfactory reproducibility in tests conducted by different laboratories, the material may be resampled and retested when requested by either the manufacturer or the purchaser. This may apply in instances when the first test results do not conform to the requirements prescribed in these specifications. The final result to be used shall be the average of at least two sets of results, each of which has been obtained by following in detail the specified testing procedures.

Methods of Testing

6. The properties enumerated in these specifications shall be determined in accordance with the following methods of test:

(*a*) *Particle Size.*—Methods of Test for Sieve Analysis and Water Content of Refractory Materials (A.S.T.M. Designation: C 92) of the American Society for Testing Materials,[3] using the procedure for wet sieve analysis described in Section 5 (*b*).

(*b*) *Water Content.*—Methods C 92, using the procedure for water content described in Section 4 (*a*).

(*c*) *Bonding Strength.*—Five 9-in. straight fireclay brick having plane surfaces and true rectangular shape shall be used as specimens for this test. They shall have an average modulus of rupture of not less than 600 psi. when determined in accordance with the Methods of Test for Cold Crushing Strength and Modulus of Rupture of Refractory Brick and Shapes (A.S.T.M. Designation: C 133) of the American Society for Testing Materials.[3] Each of the 5 brick shall be cut into two equal parts on a plane parallel to the $2\frac{1}{2}$ by $4\frac{1}{2}$ in. face. The uncut $2\frac{1}{2}$ by $4\frac{1}{2}$ in. face of the brick shall be used when forming the 5 test joints. The test mortar shall be made to troweling consistency. Approximately double the quantity required to form a joint $\frac{1}{16}$ in. in thickness shall be placed on the uncut face of one half-brick. Two spacing rods consisting of drill rods 4 in. in length and $\frac{1}{16}$ in. in diameter shall be placed in the mortar parallel to the $2\frac{1}{2}$ in. edge of the brick and $\frac{3}{4}$ in. from that edge. The uncut face of the other half-brick shall then be placed on the mortar. The excess mortar in the joint shall be forced out by pressing on the top half-brick and at the same time moving it with a to-and-fro motion in the direction of the $4\frac{1}{2}$ in. dimension of the brick, using the spacing rods as rollers. A total of 5 movements in each direction shall be given, and the distance of travel for the top half-brick shall be $\frac{1}{4}$ in. from center in each direction. The rods shall then be withdrawn and the excess mortar cleaned from the surface of the joint. The 5 prepared joints shall be air-dried undisturbed at room temperature for 24 hr., and then oven-dried at 220 to 230 F. (105 to 110 C.) for 18 hr. (overnight). Upon removal from the oven, they shall be allowed to cool by radiation until cool to the touch (about 6 hr.), at which time they shall be tested for modulus of rup-

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

ture in accordance with Methods C 133. In conducting this test, the upper bearing edge shall be brought to bear on the joint itself. The average strength of 5 joints shall be reported.

(*d*) *Refractoriness.*—A pier consisting of two whole and two half-brick laid flat shall be laid up with test mortar made to troweling consistency. Nine-inch fire-clay brick shall be used for this purpose and shall be of suitable refractoriness to withstand the heat treatment they will receive during testing, that is, the brick to be used for the intermediate heat duty class of mortar shall have a P.C.E. not lower than 29; for the high heat duty mortar, a P.C.E. not lower than 31–32; and for the super duty mortar, a P.C.E. not lower than 33. The top and bottom brick in the pier shall be the two whole brick. Between these, the two half-brick shall be placed so that the original ends and the mortar will form a vertical joint. Joints of uniform thickness in the pier shall be made with the aid of $\frac{3}{32}$-in. drill rod spacers, 6 in. in length. Mortar shall be applied to the large face of one of the whole brick, after which four spacing rods shall be placed across it $\frac{1}{2}$ in. from the ends and $\frac{1}{2}$ in. either side of the center. A large face and the original end of a half-brick shall be coated with mortar and the coated face placed in contact with the rods on the lower brick. The other half-brick shall be prepared in the same manner, and when putting it in position to complete the vertical joint, a spacing rod shall be placed across and in the center of that joint. Four rods shall also be used to form the top horizontal joint in the manner prescribed for the bottom horizontal joint. Mortar in excess of that actually required to form the joints shall be used. The rods shall be adjusted so that their ends are flush with one side of the pier. The brick units shall be firmly pressed or rubbed together so that the joints formed shall not exceed $\frac{1}{8}$ in. in thickness. The excess mortar shall be carefully removed from the joints, particularly on the side where the rods are flush. The spacing rods shall be removed as soon as the mortar has developed an initial set, which prevents bulging of the mortar from the joint. The pier shall be air-dried undisturbed at room temperature for 24 hr., and then oven-dried at 220 to 230 F. (105 to 110 C.) for 18 hr. (overnight). The pier shall then be heated to the temperature prescribed for the class of mortar being tested. The heating shall be carried out in accordance with all applicable portions of the Method of Test for Permanent Linear Change after Reheating of Refractory Brick (A.S.T.M. Designation: C 113) of the American Society for Testing Materials.[3] When testing the intermediate heat duty mortar, Schedule B as specified in Table I of Method C 113 shall be used. When testing high heat duty mortar, Schedule C as specified in Table I of Method C 113 shall be used, with the exception that when reaching 2730 F. (1500 C.) after 3 hr., that temperature shall be maintained for a period of 5 hr. The super duty mortar shall be tested in accordance with Schedule C as specified in Table I of Method C 113. After the test, the pier shall be examined for evidence of the mortar having flowed out of the joints.

Tentative Classification of

INSULATING BACK-UP BLOCK AND INSULATING FIRE BRICK[1]

A.S.T.M. Designation: C 155 - 41 T

ISSUED, 1940; REVISED, 1941.

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. This classification pertains to two kinds of heat-insulating materials, each of which is intended for different service conditions. The materials included in the broad classification of insulating fire brick are divided into five specific groups.

Insulating Back-Up Block

2. *Insulating Back-Up Block.*—A shaped product having a very low thermal conductivity and a bulk density of less than 30 lb. per cu.ft., suitable for back-up insulation of industrial furnaces.

Insulating Fire Brick

3. (*a*) *Insulating Fire Brick.*—A fire brick having a low thermal conductivity and a bulk density of less than 70 lb. per cu.ft., suitable for lining industrial furnaces.

(*b*) *Groups of Insulating Fire Brick.*—The grouping of insulating fire brick in accordance with Table I is based upon bulk density (weight per cubic foot) and the behavior in the permanent linear change test conducted at the specified temperature.

TABLE I.—GROUPING OF INSULATING FIRE BRICK.

Group Identification	Permanent Linear Change Not More Than 2 Per Cent When Tested At	Bulk Density Not Greater Than
Group 16.....	1550 F. (845 C.)	34 lb. per cu.ft.
Group 20.....	1950 F. (1065 C.)	40 lb. per cu.ft.
Group 23.....	2250 F. (1230 C.)	48 lb. per cu.ft.
Group 26.....	2550 F. (1400 C.)	52 lb. per cu.ft.
Group 28.....	2750 F. (1510 C.)	60 lb. per cu.ft.

Methods of Testing

4. The properties enumerated in this classification shall be determined in accordance with the following methods of test of the American Society for Testing Materials:

(*a*) *Bulk Density (Weight per Cubic Foot).*—Methods of Test for Size and Bulk Density of Refractory Brick (A.S.T.M. Designation: C 134).[2]

(*b*) *Permanent Linear Change.*—Methods of Testing Insulating Fire Brick (A.S.T.M. Designation: C 93), Sections 10 to 14.[2] For the purpose of this classification, the percentage of permanent linear change shall be obtained from only the 9-in. dimension of the test brick.

[1] Under the standardization procedure of the Society, this classification is under the jurisdiction of the A.S.T.M. Committee C-8 on Refactories.

[2] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Tentative Method of

PANEL TEST FOR RESISTANCE TO THERMAL AND STRUCTURAL SPALLING OF FIRECLAY PLASTIC REFRACTORIES[1]

A.S.T.M. Designation: C 180 - 43 T

ISSUED, 1943.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. This method of test covers the procedures for determining the resistance of high heat duty and super duty fireclay plastic refractories to the separate and combined effects of structural and thermal spalling. This method makes use of the general requirements and procedures prescribed in the Method of Panel Test for Resistance to Thermal and Structural Spalling of Refractory Brick (A.S.T.M. Designation: C 38) of the American Society for Testing Materials,[3] and supplements that method by giving the detailed requirements of the procedures to be followed for testing high heat duty and super duty fireclay plastic refractories. The procedures to be followed for the two types of plastic refractories differ solely in the temperature of preheating the panels, as prescribed in Section 5 of this method.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Power Press.*—A power press (preferably of the hydraulic type) equipped with a suitable mold (Note) for forming a 9-in. brick and capable of exerting a total load of about 80,000 lb.

NOTE.—It is advisable to have the mold oversize so that after drying the brick will be close to 9 by 4½ by 2½ in. in size.

(*b*) *Spalling Apparatus.*—Panel spalling apparatus as prescribed in Method C 38.[3]

Test Specimens

3. (*a*) *Number of Specimens.*—The 14 test specimens shall consist of 9 by 4½ by 2½-in. brick each prepared by molding the plastic refractory material in a power press.

(*b*) *Molding of Specimens.*—A power press shall be used to mold the test specimens and the pressure shall be applied to the 9 by 4½ in. area of the brick. In order to facilitate filling the

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-8 on Refractories.

[2] Accepted by Committee E-10 on Standards, March 20, 1943.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

mold, the plastic refractory material shall be broken into pieces varying in size, the largest being about $1\frac{1}{2}$ in. in size. When these pieces are pressed into the mold, care shall be taken to obtain uniform packing and distribution of the material. A pressure of approximately 1000 psi. is usually high enough to form a satisfactory test specimen. The pressure used shall be sufficient to cause the molded mass to be of uniform density and structure throughout and without voids. The pressure shall not, however be so high that the plastic portion of the mix is forced out of the mold by extrusion through the clearance space (not over $\frac{1}{16}$ in.) between the plunger and the walls of the mold. The same pressure in pounds per square inch shall be used for molding each of the specimens.

(*c*) *Drying of Specimens.*—The specimens shall be dried for 24 hr. at a temperature of 220 to 230 F. (105 to 110 C.), or until they contain not more than 1.0 per cent water.

Procedure

4. The procedures shall be in general as outlined in Method C 38,[3] supplemented by the details for preheating and spalling as prescribed in the following Sections 5 and 6.

Preheating Test Panels

5. (*a*) The test brick shall be laid up with kaolin mortar having a pyrometric cone equivalent not below cone No. 34.

(*b*) The test panels and dummy brick shall be insulated with group 26 insulating fire brick weighing 46 ± 2 lb. per cu. ft.

(*c*) For high heat duty plastic refractories the temperature of the panel face shall be maintained at an average temperature of 2910 F. (1600 C.) for a period of 24 hr.

(*d*) For super duty plastic refractories the temperature of the panel face shall be maintained at an average temperature of 3000 F. (1650 C.) for a period of 24 hr.

(*e*) The furnace gas pressure during the 24-hr. preheating period shall be within the limits of 0.4 to 0.5 in. of water for gas-fired furnaces.

Spalling Procedure

6. (*a*) The panel insulation shall be removed prior to spalling.

(*b*) The gas-fired furnace shall be operated so as to obtain a temperature of 2550 F. (1400 C.) before the panels are cooled by means of the blast.

(*c*) The period for heating shall be 10 min. and for cooling 10 min., making a 20-min. cycle.

(*d*) The cooling shall be accomplished by means of an air-water mist blast, which shall be delivered through a vertical manifold with an opening measuring $3\frac{1}{2}$ by 24 in., as shown in Fig. 6 of Method C 38.[3] Air shall be delivered through each cooling unit at the rate of 1400 cu. ft. per min. The water used in the four spray units shall be filtered, and measured by means of a meter. The spray nozzles shall be regulated to deliver $2\frac{1}{2}$ gal. of water during the first 8 min. of the cycle. No water shall be used during the last 2 min. The cooling units shall be operated with a reciprocating motion so as to pass back and forth across the surface of the entire panel, including the dummy guard brick, 125 times in 10 min.

(*e*) The spalling treatment shall consist of 12 cycles, after which the furnace shall be shut off and the panels given two additional cooling cycles without the water mist.

(*f*) After completion of the spalling treatment the test brick shall be removed from the panel and weighed to the nearest 0.05 lb. After removal of the spalls, the brick shall be reweighed and the loss calculated.

Tentative Method of Test for

COMBINED DRYING AND FIRING SHRINKAGE OF FIRECLAY PLASTIC REFRACTORIES[1]

A.S.T.M. Designation: C 179 - 43 T

ISSUED, 1943.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. This method of test covers a procedure for determining the combined drying and firing shrinkage of high heat duty and super duty fireclay plastic refractories.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Power Press.*—A power press (preferably of the hydraulic type) equipped with a suitable mold (Note) for forming a 9-in. brick and capable of exerting a total load of about 80,000 lb.

NOTE.—It is advisable to have the mold oversize so that after drying the brick will be close to 9 by 4½ by 2½ in. in size.

(*b*) *Drying Oven.*—An oven preferably of a size to hold six 9-in. brick.

(*c*) *Kiln.*—A down-draft type kiln of such design that the flame, as coming directly from the burner, cannot impinge upon the test specimens.

(*d*) *Rule.*—A 12-in. steel rule,[3] one edge graduated in 0.02-in. increments. A hooked rule is convenient to use, and a suitable type is described in Section 2 (*a*) of the Methods of Test for Size and Bulk Density of Refractory Brick (A.S.T.M. Designation: C 134) of the American Society for Testing Materials.[4]

Test Specimens

3. (*a*) *Number of Specimens.*—Six specimens molded from the plastic refractory material will be required. Three specimens shall be used for the test and three used as supporting pieces during the kiln heat treatment.

(*b*) *Molding of Specimens.*—The power press shall be used to mold the test specimens and the pressure shall be applied to the 9 by 4½ in. area of the specimen. In order to facilitate filling the mold, the plastic refractory material shall be broken into pieces varying in

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-8 on Refractories.

[2] Accepted by Committee E-10 on Standards, March 20, 1943.

[3] The Lufkin rule No. 2603 R or equivalent is satisfactory.

[4] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

size, the largest being about 1½ in. in size. When these pieces are pressed into the mold, care shall be taken to obtain uniform packing and distribution of the material. A pressure of approximately 1000 psi. is usually high enough to form a satisfactory test specimen. The pressure used shall be sufficient to cause the molded mass to be of uniform density and structure throughout and without voids. The pressure shall not, however, be so high that the plastic portion of the mix is forced out of the mold by extrusion through the clearance space (not over $\frac{1}{16}$ in.) between the plunger and the walls of the mold. The same pressure in pounds per square inch shall be used for molding each of the specimens. When a molded specimen is removed from the mold, it shall be placed on its 9 by 4½ in. face in its final position on the pallet or support which is to be used during drying.

(*c*) *Measurement of Specimens.*—Immediately upon placing the specimen on its support for drying, it shall be measured by means of the steel rule for its length along the center of the 9 by 4½ in. face. A reference mark (using ceramic paint) shall be made to indicate the exact position where the measurement was taken. The length shall be measured on each of the three test specimens to the nearest 0.02 in.

(*d*) *Drying of Specimens.*—The six specimens shall be dried for 24 hr. in the oven heated to a temperature of 220 to 230 F. (105 to 110 C.), or until they contain not more than 1.0 per cent of water.

Procedure

4. (*a*) *Placing Specimens in Kiln.*—The dried specimens shall be placed in the kiln in accordance with Section 4 of the Method of Test for Permanent Linear Change after Reheating of Refractory Brick (A.S.T.M. Designation: C 113) of the American Society for Testing Materials,[4] with the exception that the supporting brick shall consist of the three plastic refractory brick prepared for that purpose (see Section 3 (*a*)).

(*b*) *Temperature Measurements.*—Temperature measurements shall be conducted in accordance with Section 5 of Method C 113.

(*c*) *Test Temperature Schedules.*—The kiln shall be operated at the heating schedule for the class of refractory being tested as follows:

(*1*) High heat duty fireclay plastic refractories shall be heated in accordance with Schedule B as specified in Table I of Method C 113.

(*2*) Super duty fireclay plastic refractories shall be heated in accordance with Schedule C as specified in Table I of Method C 113.

(*d*) *Remeasuring Specimens.*—After completion of the heat treatment, the closed kiln shall be allowed to cool for a period of at least 10 hr. or until the test specimens are cool enough to handle. The three test specimens shall then be remeasured in accordance with Section 3 (*c*).

Calculation and Report

5. The combined drying and firing shrinkage expressed as a percentage shall be calculated on the basis of the original plastic length of the specimen, and the average value for the three specimens shall be reported.

Tentative Method of Test for

WORKABILITY INDEX OF FIRECLAY PLASTIC REFRACTORIES[1]

A.S.T.M. Designation: C 181 – 43 T

ISSUED, 1943.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. This method of test covers a procedure for obtaining the workability index of fireclay plastic refractory material by measuring the plastic deformation of a molded test specimen when subjected to impacts. The data obtained serve as a measure of the facility with which plastic refractory material can be rammed or pounded into place in a furnace.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Rammer.*—A device known as the sand rammer[3] (see Fig. 1), or an apparatus capable of performing the same operation. It shall consist essentially of a steel cylindrical mold 2.00 in. in inside diameter and 4.75 in. in length, supported in a vertical position on the same axis as a shaft to which shall be fastened a plunger that fits inside the mold. A 14-lb. cylindrical weight slides on the same shaft and shall be arranged so that it can be made to fall a distance of 2 in. before engaging a collar fastened to the shaft. As shown in Fig. 1, the weight may be raised by the use of a suitable cam to which a handle is attached.

(*b*) *Rule.*—A steel rule, one edge graduated in 0.02-in. increments.

Test Specimens

3. (*a*) *Number of Specimens.*—Four cylindrical test specimens shall be molded from the sample of plastic refractory material.

(*b*) *Molding of Specimens.*—To facilitate filling the mold, the sample shall be broken into pieces varying in size, the largest being about 1 in. in size. Three hundred grams of the sample shall be used for molding each test

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-8 on Refractories.

[2] Accepted by Committee E-10 on Standards, March 20, 1943.

[3] This apparatus is available from the Harry W. Dietert Co., 9330 Roselawn Avenue., Detroit, Mich.

specimen. After placing the material in the mold, it shall be subjected to 10 impacts by turning the handle, which causes the weight to be raised 2 in. and then dropped upon the collar attached to the plunger shaft. The mold shall then be upended and an additional 10 impacts given the specimen. The molded test specimen shall then be extruded from the mold by the use of a suitable auxiliary plunger.

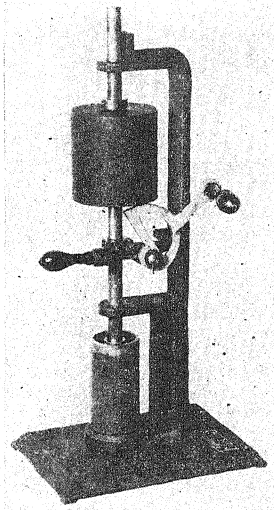


FIG. 1.—Apparatus for Workability Index Test.
The impacts applied to the test specimen during molding in the mold are obtained by turning the handle, which causes the weight to be raised 2 in. and then dropped upon the collar attached to the plunger shaft.

Procedure

4. Upon removing the cylindrical test specimen from the mold, the length shall be measured with the steel rule to the nearest 0.02 in. Four measurements shall be taken, one at each quadrant, and the average of the four measurements shall be used. The test specimen shall then be placed in the apparatus in the same position that it assumed while being molded. The plunger shall be allowed to rest on the top end of the test specimen. The distance the vertical shaft projects above the surface of the top bearing of the apparatus shall then be measured to the nearest 0.02 in. Three impacts shall be given the test specimen. The projection of the shaft shall be remeasured and the difference in inches between the two measurements recorded as the deformation of the test specimen.

Calculation and Report

5. The percentage deformation on the basis of the original length shall be calculated for each of the four test specimens and the average value reported as the workability index. The report shall also state whether any test specimen crumbled as a result of the three impacts.

Tentative Method of Test for

THERMAL CONDUCTIVITY OF INSULATING FIRE BRICK[1]

A.S.T.M. Designation: C 182 - 43 T

ISSUED, 1943.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. This method of test outlines the general procedure for determining the comparative thermal conductivity of insulating fire brick under standardized conditions of testing.[3]

Apparatus

2. The apparatus shall conform in close detail with that shown in the approved drawings.[4] The equipment is shown in Figs. 1 and 2 and the essential parts are:

(*a*) *Heating Chamber.*—A heating chamber, shown in Fig. 3, shall be capable of being heated electrically over a temperature range from 400 to 2800 F. in a neutral or oxidizing atmosphere. The temperature of the heating unit shall be controlled by a mechanism capable of maintaining the temperature in the chamber constant to within plus or minus 5 F.

(*b*) *Calorimeter Assembly.*—A copper calorimeter assembly, of the design shown in Fig. 4, shall be used for measuring the quantity of heat flowing through the test specimen. The water circulation is such that adjacent passages contain incoming and outgoing streams of water. The calorimeter shall be 3 by 3 in. square and shall have one inlet and one outlet water connection. The inner guard surrounding the calorimeter shall be 9 by 13½ in. and shall have two inlet and two outlet water connections. The outer guard shall extend 2 in. laterally from the inner guard and shall extend vertically to the member comprising the bottom of the heating chamber (see Fig. 3). The separation between the calorimeter and the inner guard shall be $\frac{1}{32}$ in.

(*c*) *Water-Circulating System.*—A water-circulating system for supplying the calorimeter assembly with water at constant pressure and at a temperature which is not changing at a rate greater than 1 F. per hr. shall be provided. The inlet water pressure shall be at least the

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-8 on Refractories.

[2] Accepted by Committee E-10 on Standards, April 15, 1943.

[3] The committee is investigating another method of test which may be adopted as an alternative method at a later date.

[4] The complete set of drawings necessary for the construction of the apparatus requires too much space to be included with this method, but is available at a nominal charge from the Babcock & Wilcox Co., Refractories Division, Technical Dept., 85 Liberty St., New York, N.Y.

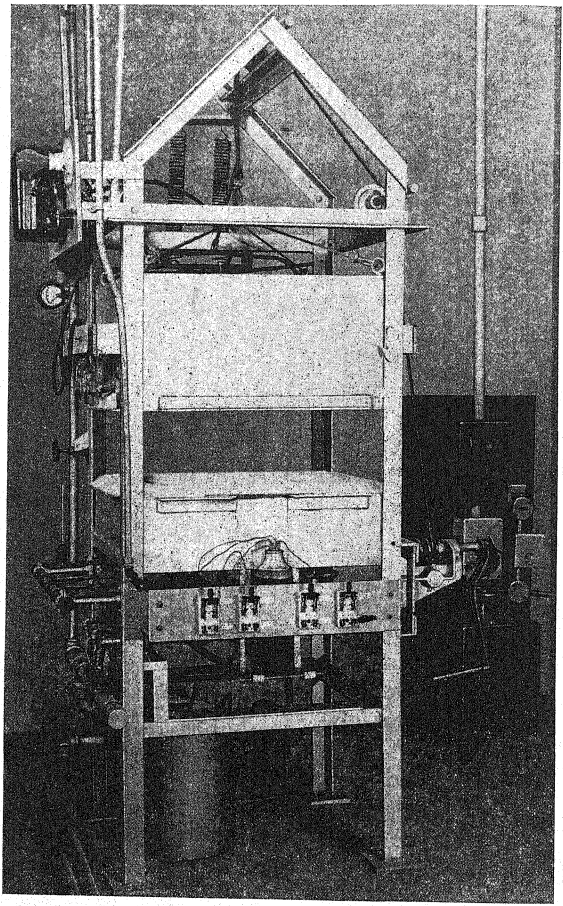


FIG. 1.—Photograph of Thermal Conductivity Apparatus.
The upper half of the heating chamber has been raised so as to introduce the test samples.

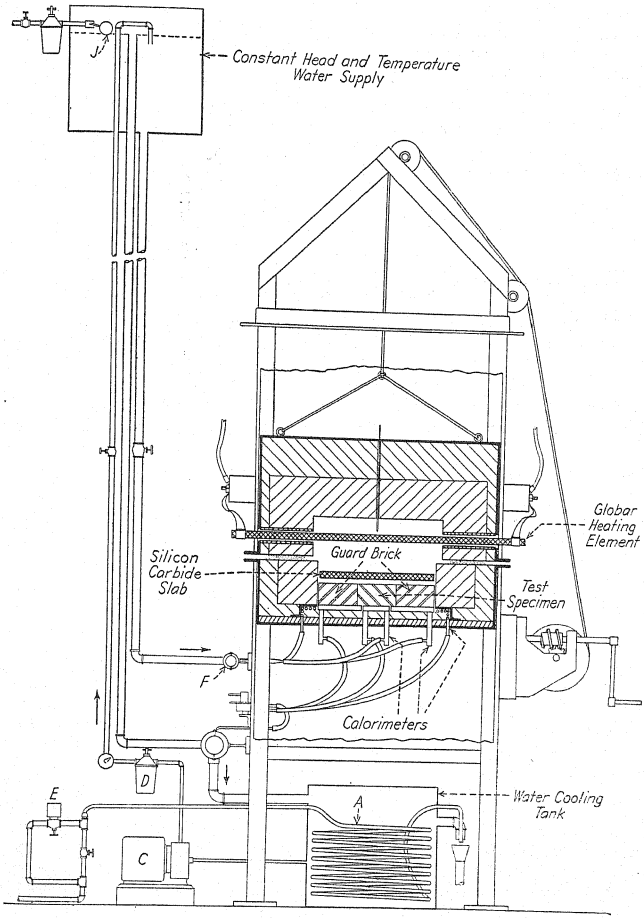


FIG. 2.—Diagram Showing Essential Parts of Thermal Conductivity Apparatus.

A—Cooling Coil.
C—Water Pump.
D—Water Filter.
E—Magnetic Control Valve.
F—Thermostat.
J—Float Valve.

equivalent of 10 ft. of hydrostatic pressure. The inlet water temperature shall at all times be within plus or minus 5 F. of the room temperature. Figure 5 shows the arrangement that shall be used for meeting these conditions. The regulating valves for controlling the rate of water flow through the calorimeter assembly shall be capable of maintaining a constant flow rate within plus or minus 1 per cent during the test period.

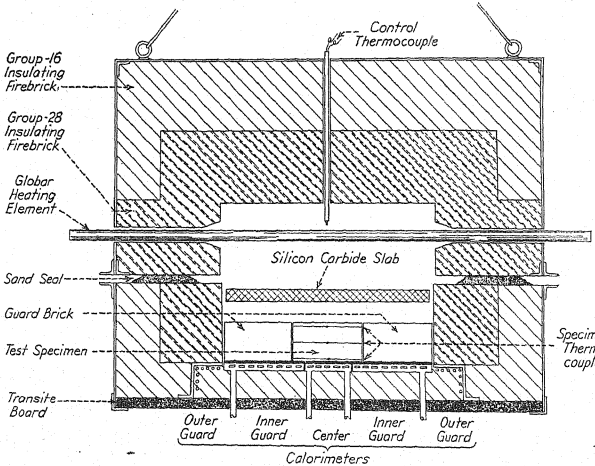


FIG. 3.—Diagrammatic Section Through Heating Chamber.

(*d*) *Instruments for Measuring Temperature of Specimen.*—Thermocouples shall be embedded in the test specimen at three points for measuring the temperature. Chromel-alumel thermocouples shall be used for temperatures below 1400 F., and above that temperature platinum-platinum 10 per cent rhodium thermocouples shall be used. The platinum thermocouples may also be used at the lower temperatures, but the emf. would not be as high as when using base-metal thermocouples. Wire of No. 28 gage shall be used for making either type of thermocouple. The emf. for the temperature readings shall be taken with a potentiometer having an instrument error of not more than plus or minus 0.05 mv., and the cold junctions of the thermocouples shall be immersed in a mixture of ice and water.[5]

(*e*) *Instrument for Measuring Temperature Rise in Calorimeter Water.*—A multiple differential thermocouple shall be used for measuring within an

[5] It is recommended that the procedure described in "Thermoelectric Thermometry," by W. F. Roeser, National Bureau of Standards, and "Methods of Testing Thermocouples and Thermocouple Materials," by W. F. Roeser and H. T. Wensel, National Bureau of Standards, be followed. These papers are contained in "Temperature, Its Measurement and Control" published by the Reinhold Publishing Corp., 330 W. 42nd St., New York 18, N. Y.

accuracy of not less than 1 per cent the temperature rise of the water flowing through the calorimeter. The thermocouple shall be immersed at least $3\frac{1}{2}$ in. in the inlet and outlet connections, and

(*f*) *Instruments for Measuring Temperature Difference between Calorimeter and Inner Guard.*—Differential 10X copper-constantan thermocouples shall be located in the calorimeter and inner

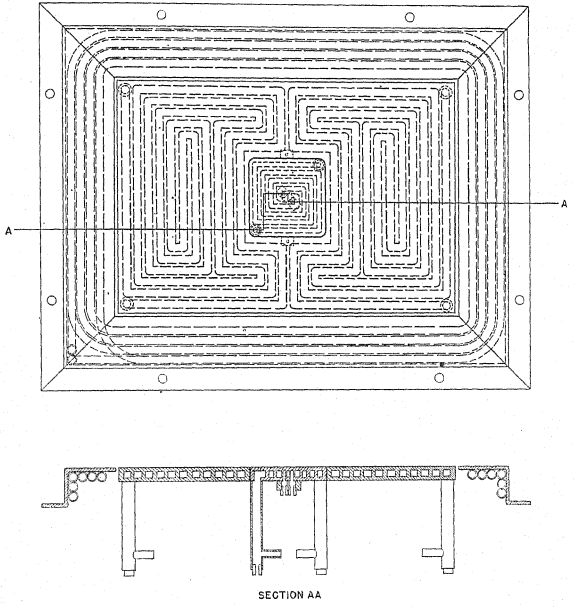


FIG. 4.—Design of Calorimeter and Guard Rings.

the junctions shall be not more than $\frac{1}{4}$ in. distant from the bottom of the calorimeter. A differential 10X copper-constantan thermocouple shall be used and the millivolt readings shall be taken with a potentiometer having an instrument error of not more than plus or minus 0.01 mv. in the range between 0 to 2 mv.

guard for measuring the temperature differences between the calorimeter and inner guard. (The temperature difference during a test shall be maintained at a value less than plus or minus 0.05 F.) The thermocouple junctions shall be placed in the four wells provided for that purpose, and millivolt readings shall

be taken with a potentiometer having an instrument error of not more than plus or minus 0.01 mv. in the range between 0 to 2 mv.

Test Specimen and Guard Brick

3. The sample shall consist of three 9-in. straight brick which are representative of the material being tested. These brick shall be selected for uniformity of structure and bulk density, and they shall be free from broken corners or edges. One brick shall be used as the test specimen, and one each of the other two brick shall be used as guard brick on either side of the specimen to prevent side flow of heat. The test specimen and guard brick shall cover an area of approximately 9 by $13\frac{1}{2}$ in.

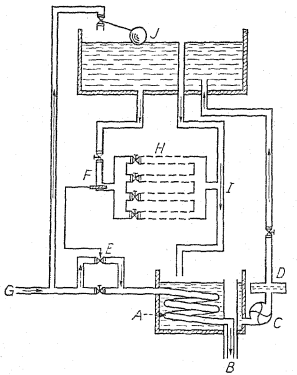


FIG. 5.—Water-Circulating System with Automatic Temperature Control.
A—Cooling Coil.
B—Connection to Drain.
C—Water Pump.
D—Water Filters.
E—Magnetic Control Valve.
F—Thermostat.
G—Water Inlet.
H—Calorimeter and Guards.
I—Overflow Pipe.
J—Float Valve.

Preparation of Test Specimen and Guard Brick

4. (*a*) The 9 by $4\frac{1}{2}$ in. faces of the three brick shall be ground flat and parallel and the thickness shall not vary more than plus or minus 0.01 in. The thickness shall be not more than 3 nor less than 2 in. The sides that are to be placed in contact shall be ground flat and at right angles to the 9 by $4\frac{1}{2}$-in. face.

(*b*) The test specimen shall be dried at 220 to 230 F. for 12 hr., after which time its bulk density, in pounds per cubic foot, shall be determined in accordance with the Methods of Test for Size and Bulk Density of Refractory Brick (A.S.T.M. Designation: C 134) of the American Society for Testing Materials,[6] with the exception that the thickness measurement shall be made in accordance with the Note of Section 5 (*b*) in those methods.

(*c*) Holes for the thermocouple wires shall be drilled through the $4\frac{1}{2}$-in. dimension of the test specimen by the use of a drilling jig so as to obtain accurate placement of the thermocouples. The three thermocouples shall be located so that the hot junction of the first couple is 0.02 in. below the hot face of the test specimen, the junction of the second at the mid point, and the junction of the third 0.02 in. above the cold face (see Fig. 3). The thermocouple wires leading out from the hot junctions shall be located in planes parallel to the calorimeter surface. In order to have the hot junctions over the center of the calorimeter, they shall be located on an axis passing through the center of and at right angles to the 9 by $4\frac{1}{2}$ in. area of the test specimen.

Procedure

5. (*a*) Two strips of asbestos paper $13\frac{1}{2}$ in. in length by $\frac{1}{2}$ in. in width by 0.02 in. in thickness shall be placed along

[6] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

the 13½-in. dimension of the inner guard at the outside edges. These strips act as spacers to prevent contact between the brick and the calorimeter assembly. The test specimen shall then be placed centrally over the center calorimeter section on its 9 by 4½-in. face, and the guard brick placed at the sides of the test specimen so as to cover completely the calorimeter and inner guard area. The small space between the furnace walls and the three brick shall be filled with granulated insulating fire brick.

(*b*) The heating chamber shall then be placed in position, water started flowing through the calorimeter assembly, and current supplied to the heating unit. The rate of water flow through the calorimeter shall be maintained between 120 and 200 g. per min., and shall be determined by weighing the quantity of water collected during a measured time period. The weight of water collected shall be not less than 200 g. and shall be weighed to an accuracy of plus or minus 0.5 g. The rate of flow shall be constant within plus or minus 1 per cent during the test period.

(*c*) The furnace shall then be allowed to reach a condition of steady state of heat flow (test period), which shall be that condition when the measured flow of heat into the calorimeter varies less than 2 per cent over a 2-hr. period, during which time the temperature difference between the calorimeter and inner guard has not been more than 0.05 F., the hot face of the test specimen has not varied more than plus or minus 5 F., and the temperature of the water entering the calorimeter has not varied at a rate of more than 1 F. per hr. (Note 1). Usually, 12 hr. or more are needed to obtain a balance with the apparatus after a definite change is made in the hot-face temperature.

(*d*) After the steady state of heat flow has been reached, the temperature of the test specimen, the rate of water flow through the calorimeter, and the temperature rise of the water flowing through the calorimeter shall be measured. At least three sets of readings (Note 2) shall be taken during the 2-hr. holding period, and these shall be averaged for the final values for that particular heating chamber temperature. Such data shall be obtained when the heating chamber is maintained at the temperatures recommended in Table I. Other temperatures than those given in the table may be used to obtain additional information.

TABLE I.—HEATING CHAMBER TEMPERATURE.

Group Identification[a]	Bulk Density Not Greater Than, lb. per cu. ft.	Recommended Temperatures for Control Points		
		Low	Medium	High
Group 16.....	34	900 F.	.....	1600 F.
Group 20.....	40	900 F.	1400 F.	2000 F.
Group 23.....	48	900 F.	1500 F.	2300 F.
Group 26.....	52	900 F.	1650 F.	2600 F.
Group 28.....	60	900 F.	1750 F.	2800 F.

[a] See the Classification of Insulating Back-Up Block and Insulating Fire Brick (A.S.T.M. Designation: C 155) of the American Society for Testing Materials.[2]

(*e*) At the conclusion of the test, the specimen shall be removed and cut in two, close to the thermocouple junctions. The specimen shall then be ground to expose the hot junctions, and the distance between them shall be measured to the nearest 0.01 in. If the test specimen upon being cut in two shows abnormal internal voids or cracks, this fact shall be stated in the report as the results will not be representative of the material.

NOTE 1.—Significant errors will result if the tolerances specified are exceeded.

NOTE 2.—From these data a preliminary thermal conductivity calculation can be made, using estimated distances between thermocouple junctions in the test specimen.

Record of Test Data

6. The following data shall be recorded, and items (*3*) through (*7*) shall

be recorded for each 2-hr. test period (steady state of heat flow):

(*1*) Linear dimensions of test specimen,

(*2*) The distance between thermocouple junctions located in the test specimen,

(*3*) Three sets of temperature readings as measured by the three thermocouples in the test specimen,

(*4*) The mean temperature at each thermocouple in the test specimen as calculated from the temperatures recorded in item (*3*),

(*5*) The average rise in temperature of the water flowing through the calorimeter,

(*6*) The average rate of water flow through the calorimeter, and

(*7*) The rate of heat flow through the test specimen per unit area.

Calculations[7]

7. The thermal conductivity shall be calculated as follows:

$$k = \frac{qL}{A(t_1 - t_2)}$$

where:

k = thermal conductivity in Btu. per in. per sq. ft. per hr. per deg. Fahr.,

q = the Btu. per hr. flowing into the calorimeter (temperature rise in degrees Fahrenheit of the water flowing through the calorimeter times the weight of water in pounds flowing per hour),

L = thickness in inches (distance between hot junctions at which t_1 and t_2 are measured),

t_1 = higher of two temperatures measured in the test specimen, in degrees Fahrenheit,

t_2 = lower of two temperatures measured in the test specimen, in degrees Fahrenheit, and

A = area of center calorimeter in square feet.

Report

8. The report shall include the following:

(*a*) Brand name or other identifying information,

(*b*) Bulk density of the dried test specimen (pounds per cubic feet),

(*c*) General description of the test specimen before and after test with respect to possible structural changes caused by exposing the test specimen to the heating chamber temperatures,

(*d*) The thermal conductivity calculated in accordance with Section 7 at the mean temperatures specified in Section 5 (*d*) and reported at the mean of the two temperatures used in the calculation,

(*e*) A curve showing the actual thermal conductivity values obtained *versus* mean temperatures, and

(*f*) When requested, the data recorded for Section 6 shall be included in the report.

[7] See the Symbols for Heat Transmission (A.S.T.M. Designation: C 108), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Tentative Methods of Test for

SIEVE ANALYSIS AND WATER CONTENT OF REFRACTORY MATERIALS[1]

A.S.T.M. Designation: C 92 - 43 T

ISSUED, 1943.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. (*a*) These methods of test cover procedures for a wet method and a dry method for sieve analysis of refractory materials, and also procedures for determining the water content of refractory materials in the wet condition.

(*b*) *Wet Sieve Analysis.*—Water promotes the slaking of clays and helps to separate fine particles and to wash them from the larger grains. Consequently, this method will provide more reliable information than the dry method on materials to which water is added and in which slaking would occur in normal industrial use. This method is recommended, therefore, for use with such materials.

(*c*) *Dry Sieve Analysis.*—The dry method is not as effective as the wet method in determining the amount of material present in the smaller particle sizes. It is recommended (*1*) for clays only when the slaking action of water is undesirable, (*2*) when the material is in the form of coarsely ground grog and calcine, and (*3*) when the clay is to be used in such a way that the size of the ultimate particles is of secondary importance.

(*d*) *Water Content.*—There is included a method for determining the water content of air-dry samples as received, so that the sieve analysis can be calculated on the dry basis. There is also included a method for obtaining the water content of other refractory materials, such as plastic refractories and wet mixes.

Sieves

2. Sieves of appropriate mesh from either list given in Table I shall be used. The wire cloth for the sieves[3] shall be woven (not twilled) and shall be mounted without distortion or looseness in circular frames 8 in. in diameter. Pans and covers shall be provided for the sieves.

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[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee C-8 on Refractories.

[2] Accepted by Committee E-10 on Standards, March 20, 1943.

Prior to their present publication as tentative, these methods were published as tentative from 1932 to 1936, being revised in 1934. They were adopted in 1936, but were withdrawn, revised, and republished as tentative in 1943.

[3] See the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

TABLE I.—A.S.T.M. SIEVES AND EQUIVALENT TYLER STANDARD SIEVES.

A.S.T.M. Sieves (U. S. Standard Sieve Series Number)	Tyler Standard Sieves (Mesh Number)
No. 4	4
No. 6	6
No. 8	8
No. 12	10
No. 16	14
No. 20	20
No. 30	28
No. 40	35
No. 50	48
No. 70	65
No. 100	100
No. 140	150
No. 200	200

Sample

3. The sample submitted shall be representative of the material to be tested, and shall weigh at least four to five times the required weight of the actual test sample. Material of which the water content is to be determined shall be packed in a water-tight container.

Water Content

4. (*a*) *Wet Type Air-Setting Refractory Mortars.*—The material shall be sampled immediately after opening the original container and after carefully mixing the contents. The test samples shall weigh approximately 50 g. To facilitate handling the test sample, it is recommended that it be placed on a tared piece of waxed paper or tared aluminum weighing dish. The exact weight of the test sample shall be determined to the nearest 0.1 g. both before and after drying for 24 hr. at 220 to 230 F. (105 to 110 C.). The percentage of water shall be calculated on the as-received basis to the nearest 0.1 per cent.

(*b*) *Materials Other Then Wet Type Air-Setting Refractory Mortars* (such as ground fireclays, fireclay mortars, dry type air-setting mortars, plastic refractories, and similar materials).—If the material is shipped in the wet condition, care shall be taken that no loss of water occurs before the test sample is taken. The test sample shall weigh approximately 250 g. The exact weight of the test sample shall be determined to the nearest 0.1 g. both before and after drying for 3 hr. at 220 to 230 F. (105 to 110 C.). The percentage of water shall be calculated on the as-received basis to the nearest 0.1 per cent. The dried test sample may be required for further use (see Section 5 (*a*) and (*b*) and Section 6 (*a*) and (*b*).

Wet Sieve Analysis

5. (*a*) *Dry Materials.*—If the material is received in the dry condition, the test sample (Note 3) shall consist of the dried and weighed test sample prepared in accordance with Section 4 (*b*).

(*b*) *Wet Materials.*—Materials prepared with water (plastic refractories, wet type high-temperature bonding mortars, etc.) shall be tested as received. Two test samples shall be taken immediately upon opening the original container, and in the case of mortars after carefully mixing the contents. One sample shall be used for obtaining the water content in accordance with Section 4 (*a*) or (*b*), depending upon the type of material to be tested. The other sample (Note 3) shall be used for the sieve analysis and shall weigh approximately 250 g. It shall be weighed to the nearest 0.1 g. and then transferred to the 1-liter container (see Paragraph (*c*)). The utensils used during weighing (to which a small part of the sample may adhere) shall be washed by means of a small jet of water from a $\frac{1}{4}$-in. hose in such a manner as to insure a quantitative transfer of the weighed sample to the container.

(*c*) *Procedure.*—The test sample shall be placed in a container of about 1-liter capacity, to which sufficient water shall be added to form a slurry. Slaking shall be allowed to proceed for 1 hr., after which a further addition of water may be necessary. The test sample shall then

be transferred without loss to the finest sieve to be used in the analysis, and shall then be washed by means of a small jet of water from a $\frac{1}{4}$-in. rubber hose until the water passing through the sieve contains only traces of the sample. Care shall be exercised in the washing operation to prevent loss by splashing. It may be necessary to break up lumpy material by gentle rubbing between the fingers, but never by rubbing or pressing against the sieve. The washed test sample in the sieve shall then be dried to constant weight at 220 to 230 F. (105 to 110 C.). This usually requires about 2 hr. If desired, a preliminary drying period at a lower temperature may be used. The test sample shall then be transferred to the top or coarsest sieve of the series to be used. The sieving and weighing operations shall then be completed in accordance with Section 6 (*a*) or (*b*).

(*d*) *Calculation and Report.*—The wet sieve analysis shall be calculated for the test sample on the dry weight, and the results reported to the nearest 0.1 per cent of the material retained on each sieve (Note 4). The percentage passing the finest sieve shall be reported as the difference between 100 per cent and the sum of the percentages retained on the various sieves.

Dry Sieve Analysis

6. (*a*) *Machine Sieving.*—When the sieving is to be done by machine, the sieves shall be arranged in the order of size with the coarsest sieve at the top of the nest. The test sample (Note 3) shall consist of the dried and weighed material prepared in accordance with Section 4 (*b*). It shall be transferred to the top sieve of the nest, and shall then be subjected to the mechanical sieving operation until less than 0.1 g. of material passes through each sieve after 1 min. of sieving by hand, as described in Paragraph (*b*). The machine sieving operation usually requires about 15 min. The sieves shall then be carefully separated and the amount of material retained on each sieve shall be determined by weighing to the nearest 0.1 g.

(*b*) *Hand Sieving.*—The test sample (Note 3) shall consist of the dried and weighed material prepared in accordance with Section 4 (*b*). The sieving operation shall be carried out using one sieve at a time beginning with the coarsest sieve and from then on using successively finer sieve sizes. The sieve, with pan and cover attached, shall be alternately tapped and rotated while held in slightly inclined position so that the test sample will be well distributed over the sieve. The operation shall be continued until less than 0.1 g. of the material passes through each sieve in 1 min. of continuous sieving. The amount of material retained on each sieve shall be determined by weighing to the nearest 0.1 g.

(*c*) *Calculation and Report.*—The dry sieve analysis shall be calculated for the test sample on the dry weight, and the results reported to the nearest 0.1 per cent of the material retained on each sieve (Note 4). Dust loss shall be reported as material passing the finest sieve.

NOTE 3.—The size of the test sample may be changed by reason of the nature of the material. For example, some clays, when ground to exceedingly fine particle size, tend to pack or cake on the sieves, in which case a 100-g. sample may be used. In the case of plastic refractories, or coarsely ground mixes, the size of the sample might well be increased to 500 g. in weight.

NOTE 4.—As an alternative, the results of sieve analysis may be reported on the cumulative basis, either as the total percentage retained on or passing each sieve.

Tentative Symbols for
HEAT TRANSMISSION[1]

A.S.T.M. Designation: C 108 – 44 T

ISSUED, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

SPECIAL PRACTICES RELATING TO HEAT FLOW TERMS

Terms Ending in "ivity."—Terms ending in "ivity" designate characteristics of materials, normally independent of size or shape, sometimes called "specific properties."

Examples: Conductivity and Resistivity.

Terms Ending in "ance."—Terms ending in "ance" designate properties of a particular object, depending not only on the material, but also upon size and shape, sometimes called "total quantities."

Examples: Conductance and Transmittance.

Terms Ending in "ion."—Terms ending in "ion" designate time rate of the process of transfer; flux, flow rate.

Examples: Conduction and Transmission.

Transmission.—"Transmission," "transmissivity," "transmittance" usually refer to transfer by one or more of the processes of conduction, convection, and radiation.

Conduction.—"Conduction," "conductivity," "conductance" usually refer to transfer within a medium, and without bodily displacement as occurs with convection, and without transfer at a distance as occurs with radiation.

HEAT FLOW SYMBOLS

- A Area
- ρ Density; pounds, kilograms, etc., per unit of volume
- L Length of path of heat flow
- Q Total quantity of heat transferred (with subscripts for particular cases and to distinguish from Q for volume rate)
- Q Volume rate; discharge by volume; fluid rate of flow by volume (There also is used q with a subscript to distinguish from heat flow rate)
- c Specific heat
- T Temperature on absolute scale
- t Temperature, degrees Centigrade or Fahrenheit
- t, τ Time
- W Weight; quantity of matter measured in pounds, kilograms, etc.
- w Flow rate; pounds, kilograms, etc., per unit of time
- q Heat flow rate; time rate of heat transferred for a particular setup with any value of area A

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[1] Under the standardization procedure of the Society, these symbols are under the jurisdiction of the A.S.T.M. Committee C-8 on Refractories.

These symbols for heat transmission are identical with those appearing in the American Standard Letter Symbols for Heat and Thermodynamics Including Heat Flow (ASA No.: Z10.4-1943) of the American Standards Association.

[2] Prior to their present publication as tentative, these symbols were published as tentative from 1934 to 1939. They were adopted in 1939, published as standard from 1939 to 1944, being revised in 1941, but withdrawn, revised, and republished as tentative in 1944.

q is in general the time derivative of total quantity of heat transferred. For steady-state heat flow, q is total quantity of heat divided by time

k Thermal conductivity; heat flow rate, per unit of area, per "degree per unit of length"

$\frac{dq}{dA} = -k\frac{dt}{dL}$ general expression giving value at each point

$k = \frac{q/A}{\Delta t/L}$ for substantially uniform and steady-state heat flow through a homogeneous medium of thickness L, with plane parallel faces of area A, and a constant temperature difference Δt applied to the faces

$\frac{1}{k}$ Thermal resistivity; reciprocal of conductivity

R Thermal resistance; degrees, per unit of heat flow rate, for a particular body or setup (where the area may not be known)

$R = \frac{\Delta t}{q}$ for substantially uniform and steady-state heat flow, generally

$R = \frac{L}{kA}$ for substantially uniform and steady-state heat flow through a homogeneous medium of thickness L with plane parallel faces of area A

$\frac{1}{R}$ Thermal conductance; reciprocal of thermal resistance (C is also used)

$\frac{1}{RA}$ Thermal conductance per unit of area; heat flow, rate, per unit of area, per degree

RA Thermal resistance of unit area; degrees, per "unit of heat flow rate per unit of area." R is used for resistance for a setup with a particular area (which may not be known) and RA for resistance of unit area. In some British texts R is used for thermal resistance of unit area, here called RA

h Surface coefficient of heat transfer; heat flow rate, per unit of area, per degree, across a boundary surface

$\frac{dq}{dA} = h\Delta t$ general expression giving value at each point

$h_m = \frac{q/A}{\Delta t_m}$ for substantially uniform and steady-state heat flow, where h_m and Δt_m are constant over the area A or where one (but not both) of them varies but may substantially be represented by a mean value. h is an average property of a particular boundary condition or film and is not necessarily inversely proportional to film thickness. Δt_m is the mean absolute difference between the temperature t_s of the surface and t_f the bulk temperature of the ambient fluid, or the temperature of the surface of an adjacent solid.

U Over-all coefficient of heat transfer; sometimes called thermal transmittance per unit of area; heat flow rate, per unit of area, per degree, for a particular setup of one or more bodies and films. Then, for substantially uniform and steady-state heat flow,

$U = \frac{q/A}{\Delta t}$ Some particular area in the setup, A, may be chosen as a reference. Then for a setup of homogeneous bodies, area A_x, and adjacent films, area A_{fx}, where the corresponding values of k and h are substantially constant over the areas considered

$$\frac{1}{U} = \sum \frac{L}{kA_x/A} + \sum \frac{1}{hA_{fx}/A}$$

For parallel walls,

$$\frac{1}{U} = \sum \frac{L}{k} + \sum \frac{1}{h} \quad \text{or,}$$

$$\frac{1}{U} = \sum RA + \sum \frac{1}{h}$$

S Shape factor of a structure; heat flow rate, divided by equivalent thermal conductivity and by the temperature difference between inner and outer surfaces

$$S = \frac{q}{k\Delta t_a}$$

ϵ Total emissivity; ratio of radiant flux from a source to that for a blackbody of the same size and shape, at the same temperature, according to the Stefan-Boltzmann law which is,

$$\Phi \approx \sigma \epsilon A T^4$$

Φ Radiant flux; radiant energy per unit of time, for any area A

α Thermal diffusivity; thermal conductivity divided by heat capacity per unit volume

$$\alpha = \frac{k}{c\rho}$$

G Flow rate in pounds per unit of time, per unit of area of cross-section; called weight velocity, or mass velocity

$$G = V\rho$$

Δ Difference between values, often taken as positive when it is that difference causing flow

Tentative Definition of

THE TERM GLASS[1]

A.S.T.M. Designation: C 162 – 41 T

ISSUED, 1941.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Glass.—Glass is an inorganic product of fusion which has cooled to a rigid condition without crystallizing. Some glasses contain nonglassy material, always embedded in a glassy matrix. Glasses may be colored by suitable material in a dissolved or dispersed condition.

Glasses may be classified as:

(*a*) *One-component glasses,* consisting of single oxides, such as, silica (SiO_2), boric oxide (B_2O_3), or phosphoric oxide (P_2O_5).

(*b*) *Two-component glasses,* of which the chief commercial representatives are the soluble silicates.

(*c*) *Multi-component glasses:*

(*1*) Soda-lime-silica glasses, which include the great bulk of commercial glassware.

(*2*) Borosilicate glasses, which range from soda-lime-silica glasses containing small amounts of boric oxide to low-expansion glasses containing little or no soda or lime.

(*3*) *Special glasses:*

(*a*) "Flint" glasses, containing lead oxide (PbO), ranging from lead tubing and cut glass, to heavy lead glasses used for optical instruments and imitation jewelry.

(*b*) Optical glasses, mostly silicate or borosilicate glasses distinguished by physical perfection and a wide range of optical properties.

(*c*) Nonsilicate glasses, used for special purposes and at present of limited commercial applicability.

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[1] Under the standardization procedure of the Society, this definition is under the jurisdiction of the A.S.T.M. Committee C-14 on Glass and Glass Products.

[2] Accepted by the Society at annual meeting, June, 1941.

Tentative Specifications for

STANDARD STRENGTH CLAY SEWER PIPE[1]

A.S.T.M. Designation: C 13 – 44 T

ISSUED, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These specifications cover standard strength clay pipe intended to be used for the conveyance of sewage, industrial wastes, and storm water.

NOTE.—Attention is called to the Tentative Specifications for Extra Strength Clay Pipe (A.S.T.M. Designation: C 200)[3] and the Recommended Practice for Laying Sewer Pipe (A.S.T.M. Designation: C 12).[3]

Class

2. Pipe manufactured according to these specifications shall be known as "Standard Strength Clay Sewer Pipe."

MATERIALS AND MANUFACTURE

Materials and Manufacture

3. (*a*) Clay pipe shall be manufactured from surface clay, fire clay, or shale, or a combination of these materials.

(*b*) *Surface clay* is an unconsolidated, unstratified clay,[4] occurring on the surface.

(*c*) *Fire clay* is sedimentary clay[4] of low flux content.

NOTE.—Fire clay is usually associated with coal measures.

(*d*) *Shale* is a thinly stratified, consolidated, sedimentary clay[4] with well-marked cleavage parallel to the bedding.

(*e*) These materials or any combination thereof, when molded into pipe and subjected to suitable temperatures, shall yield a product that will be strong, durable, serviceable, free of objectionable defects, and shall conform to these specifications.

RESISTANCE TO ACTION OF ACIDS

Tests at Purchaser's Option

4. Tests for resistance to action of acids shall be optional with the pur-

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-4 on Clay Pipe.

[2] Accepted by Committee E-10 on Standards, August 28, 1944.

Prior to their present publication as tentative, these specifications were published as tentative from 1917 to 1920, being revised in 1918 and 1920. They were adopted in 1920, revised in 1924, but withdrawn and replaced in 1933 by Specifications C 13 – 33 T, which were published as tentative from 1932 to 1935, being revised in 1935. They were adopted in 1935, published as standard from 1935 to 1944, being revised in 1940, but were revised and republished as tentative in 1944.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

[4] The definitions for the terms "surface clay," "fire clay," and "shale" are based on the following definition for the term "clay":

Clay.—An earthy or stony mineral aggregate consisting essentially of hydrous silicates of alumina, plastic when sufficiently pulverized and wetted, rigid when dry, and vitreous when fired at a sufficiently high temperature.

chaser, and shall be made only when specified by him in advance or in the "call for bids." The purpose of this test is to determine the resistance of pipe to the action of acids.

Reagent

5. When testing with either hydrochloric, nitric, sulfuric, or acetic acid, as specified by the purchaser, a 1 N[5] acid solution shall be used.

Test Specimens

6. (*a*) Whenever possible, the specimens intended for these tests shall be selected from pipe that previously have been broken in the strength test. Specimens shall be sound with all edges freshly broken, free of cracks or shattered edges, about 2 in. square, weighing not more than 200 g., and shall be thoroughly cleaned with a wire brush.

(*b*) A test shall be made on at least one specimen cut from each size of pipe. The results shall be reported separately for each individual specimen.

Weighing Apparatus

7. The balance used in weighing the specimen shall be sensitive to 0.01 g. when loaded with 200 g.

Procedure

8. (*a*) The specimens to be tested shall be dried to constant weight at a temperature between 210 and 230 F.

(*b*) The specimens, upon reaching constant weight, shall be suspended and completely immersed in the test solution at a temperature between 70 and 90 F. for a period of 48 hr., then removed from the solution and carefully and thoroughly washed with hot water, allowing the washings to run into the solution in which the specimen was immersed. The solution shall be filtered and the filter washed with hot water, adding the washings to the filtrate. To the filtrate shall be added 5 ml. of H_2SO_4 (sp. gr. 1.84). The solution shall then be evaporated (avoiding loss by spattering) to about 5 ml., transferred to a porcelain crucible (previously ignited to constant weight), and heated cautiously to dryness. The residue shall then be ignited to constant weight.

Calculation

9. The percentage of acid-soluble matter shall be calculated as follows:

$$\text{Acid-soluble matter, per cent} = \frac{R}{W} \times 100$$

where:

R = weight of the residue in the porcelain crucible, and

W = weight of the dry specimen before immersion.

Acceptance or Rejection

10. When the test for resistance to action of acids is specified, the pipe of each size and shipment shall be acceptable only when the percentage of acid-soluble matter for specimens representing such pipe does not exceed 0.25 per cent.

PHYSICAL TEST REQUIREMENTS

Crushing Strength Tests

Test Specimens

11. (*a*) The specimens for purpose of tests shall be sound, full-size pipe and shall be selected by the purchaser or his representative at the point or points designated by him when placing the order.

(*b*) The manufacturer or seller shall furnish, without charge, specimens for

[5] These 1 N solutions should contain, respectively, 36.5, 63, 49, and 60 g. of the acid per liter of solution. For the purpose of these tests the solution can be prepared by taking the following volumes of acid and diluting to 1 liter: H_2SO_4 (sp. gr. 1.84), 28.5 ml.; HCl (sp. gr. 1.18), 88.9 ml.; HNO_3 (sp. gr. 1.42), 65 ml.; and glacial acetic acid (sp. gr. 1.05), 57.7 ml.

tests, up to 0.5 per cent of the number of pipe in each size of pipe furnished, except that in no case shall less than two specimens be furnished.

Measurement and Observation of Specimens

12. (*a*) The specimens shall first be freed from all visible moisture. When dry, each specimen shall be measured and inspected. The results of these observations shall be recorded.

(*b*) *Defective Specimens.*—Specimens that are observed to have fire cracks or other defects in form or dimensions, in excess of the limits permitted in these specifications, shall be discarded and replaced with additional specimens from the shipment.

Test Bearings

13. Pipe shall be tested for crushing strength by either the three-edge-bearing method or by the sand-bearing method as described in Sections 16 and 17. Either of the two specified bearing methods shall be used on retests as provided for in Section 22 (*b*) and (*c*).

Apparatus and Application of Load

14. (*a*) Any prime mover or hand power device that will apply the load at a uniform rate of about 2000 lb. per min., or in increments of not more than 100 lb. at the same rate, may be used in making the test.

(*b*) The center of the load shall be applied to the top bearing block at a point distant from the spigot end of the pipe equal to one half of the over-all length of the pipe. The test load shall be applied to the upper bearing block in such a way as to leave the bearing free to move in a vertical plane passing midway between the lower bearings. In testing pipe that is "out of straight," the lines of the bearings chosen shall be from those which appear to give most favorable conditions for fair test.

(*c*) The pipe shall be allowed to stand under load no longer than is required to apply the load and to observe and record it.

(*d*) The testing machine shall be substantial and rigid throughout, so that the distribution of the load will not be affected appreciably by the deformation or yielding of any part. The bearings shall be sufficiently rigid to transmit and receive uniform loads throughout their lengths without deflection, and shall be so attached to the machine as to transmit and receive the maximum loads produced by the tests without lost motion, vibration, or sudden shock.

(*e*) The bearings and the specimen shall be accurately centered so as to secure a symmetrical distribution of the loading on each side of the center of the pipe in every direction.

(*f*) The load shall be applied until the pipe yields by cracks passing through the shell. At the discretion of the inspector the loading may be stopped before rupture of the pipe, provided the load is at least equal to the required average strength of the pipe.

Calculation and Report

15. The crushing strength shall be calculated by dividing the breaking load or applied load as provided in Section 14 (*f*) by the net inside length of the barrel of the pipe, measuring from the bottom of the socket to the end of the spigot. The individual results of the tests of pipe of each size or each shipment shall be tabulated separately so as to show the percentage that fails to conform to the requirements of each test.

Three-Edge-Bearing Method

16. When the three-edge-bearing method is used, the ends of each specimen

of pipe shall be accurately marked in halves of the circumference prior to the test. The lower bearings shall consist of two wooden strips with vertical sides having their interior top corners rounded to a radius of approximately $\frac{1}{2}$ in. The strips shall be straight and shall be securely fastened to a rigid block at least 6 by 6 in. in cross-section. The interior vertical sides of the strips shall be parallel and spaced a distance apart of 1 in. per foot of pipe diameter, but in no case less than 1 in. Before the pipe is placed, a fillet of plaster of Paris or other equalizing material, such as sand and sulfur, thick enough to compensate for the inequalities of the pipe barrel shall be cast on and between the lower bearings. The pipe shall be placed on the fillet while the plaster of Paris is still somewhat plastic; the use of the plaster of Paris fillet may be dispensed with when agreeable to the manufacturer and the purchaser. The upper bearing shall be a rigid wooden block at least 6 by 6 in. in cross-section, straight and true from end to end. A fillet of plaster of Paris thick enough to compensate for the inequalities of the pipe barrel shall also be cast along the length of the crown. The upper bearing shall be brought in contact while the plaster of Paris is still somewhat plastic. The upper and lower bearings shall extend the full length of the pipe exclusive of the socket.

Sand-Bearing Method

17. (*a*) When the sand-bearing method is used, the ends of each specimen of pipe shall be accurately marked in quarters of the circumference prior to the test. Specimens shall be carefully bedded in sand, above and below, for one fourth the circumference of the pipe measured on the middle line of the barrel. The depth of the bedding above and below the pipe at the thinnest points shall be one half the radius of the middle line of the barrel.

(*b*) The sand used shall be clean and moist and shall be such as will pass a 4760-micron (No. 4) sieve conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11).[3] The sand in the lower bearing shall be loose when the pipe is placed.

(*c*) The top bearing frame shall not be allowed to come in contact with the pipe nor with the top bearing plate. The upper surface of the sand in the top bearing shall be struck off level with a straightedge and shall be covered with a rigid top bearing plate, the lower surface of which is a true plane, made of heavy timbers or other rigid material, capable of distributing the test load uniformly without appreciable bending. The test load shall be applied at the exact center of this top bearing plate, in such a manner as to permit free motion of the plate in all directions. For this purpose a spherical bearing is preferred, but two rollers at right angles may be used.

(*d*) The test may be made without the use of a testing machine, by piling weights directly on a platform resting on the top bearing plate; provided, however, that the weights shall be piled symmetrically about a vertical line through the center of the pipe, and that the platform shall not be allowed to touch the top bearing frame.

(*e*) The frames of the top and bottom bearings shall be made of timbers so heavy as to avoid appreciable bending by the side pressure of sand. The interior surfaces of the frames shall be dressed. No frame shall come in contact with the pipe during the test. A strip of cloth may be attached, if desired, to the inside of the upper frame on each side, along the lower edge, to prevent the escape of sand between the frame and the pipe.

Absorption Test

Test Specimens

18. (*a*) The test specimens shall be sound pieces, of the full thickness of the barrel of the pipe, with all edges broken, and may be from pipe broken in the strength tests. They shall be from 12 to 20 sq. in. in area, and shall be as nearly square as they can be readily prepared. They shall be free of observable cracks or shattered edges, and shall contain laminations and fissures only to the extent that these are representative of the pipe from which the specimens were taken.

(*b*) Each specimen shall be marked so that it may be identified with the pipe used in the strength test from which the specimen was taken. The marking shall be applied so that the pigment used shall cover not more than 1 per cent of the total superficial area of the specimen.

(*c*) One specimen shall be tested from each pipe broken in the strength test.

Weighing Apparatus

19. The balance used shall be sensitive to 0.5 g. when loaded with 1 kg., and weighings shall be read at least to the nearest gram. When other than metric weights are used, the same degree from accuracy shall be obtained.

Procedure

20. (*a*) The specimens shall be dried to constant weight in a drier or oven at a temperature of not less than 110 C. (230 F.).

(*b*) The dried specimens shall be placed in a suitable wire receptacle, packed tightly enough to prevent jostling, covered with distilled water or rain water, raised to the boiling point and boiled for 5 hr., and then cooled in water to a final temperature of 15 to 25 C. (59 to 77 F.). When cool, the specimens shall be removed from the water and allowed to drain for not more than 1 min. The superficial moisture shall then be removed by a towel or blotting paper, and the specimens immediately weighed.

Calculation and Report

21. The absorption of each specimen shall be calculated as percentage of the initial dry weight. The results shall be reported separately for each individual specimen, together with the average for all the specimens tested from the same shipment of pipe.

ACCEPTANCE OR REJECTION ON RESULTS OF TESTS

Crushing Strength and Absorption

22. (*a*) The crushing strength shall conform to the requirements prescribed in Table I and the absorption shall not exceed 8 per cent, except as provided in Paragraphs (*b*), (*c*), and (*d*).

TABLE I.—CRUSHING STRENGTH REQUIREMENTS FOR STANDARD STRENGTH CLAY SEWER PIPE.

Size, in.	Average Strength, min., lb. per linear ft.	
	Three-Edge-Bearing Method	Sand-Bearing Method
4	1000	1430
6	1000	1430
8	1000	1430
10	1100	1570
12	1200	1710
15	1400	2000
18	1700	2430
21	2000	2860
24	2400	3430
27	2750	3930
30	3200	4570
33	3500	5000
36	3900	5570

(*b*) Failure of more than 20 per cent of the specimens of a size to conform to the requirements of the strength tests or the absorption tests or both shall result in rejection of all the pipe of that size in the shipment or delivery; except that in the event of 20 per cent of the specimens in any size failing to meet the requirements, the manufacturer or seller may, with the consent of the purchaser,

furnish for test, without charge, additional specimens from the same shipment to be selected as specified in Section 11. In case more than 80 per cent of the specimens tested, including those first tested, shall show substantial conformance for each of the various tests performed, then the entire shipment or delivery for this size shall be accepted; otherwise it shall be rejected.

(*c*) However, in case of failure of pipe to conform to the strength requirements under the specified method, and the accuracy of the testing machine is questioned, a retest at the request of the manufacturer shall be made upon a machine whose accuracy is known, or the machine originally used shall be recalibrated before the retest is made.

(*d*) In addition to the requirements in Paragraph (*b*), failure of 10 per cent or more of the individual specimens tested to develop 75 per cent of the average crushing strength requirements shall be cause for the rejection of the shipment, but the manufacturer or seller may cull the pipe and submit the balance of the shipment for retest, and if the shipment then conforms to all of the requirements of these specifications it shall be accepted.

SIZES AND DIMENSIONS, WORKMANSHIP AND FINISH

Sizes and Dimensions

23. Pipe shall be furnished of the sizes and of the dimensions, including permissible variations, prescribed in Table II. When more than one length is permitted, the purchaser shall indicate, at the time of purchase, which lengths shall be furnished; unless so indicated, the manufacturer shall furnish such lengths as he may elect.

Straightness

24. Pipes intended to be straight shall have a maximum ordinate as measured from the concave side of the pipe not to exceed $\frac{1}{8}$ in. per foot of length.

Salt Glaze

25. The glaze shall consist of a continuous layer of salt glaze substantially free of large blisters or large pimples.

TABLE II.—DIMENSIONS OF STANDARD STRENGTH CLAY SEWER PIPE.

Size, in.	Laying Length		Maximum Difference in Length of Two Opposite Sides, in.	Outside Diameter of Barrel, in.		Inside Diameter of Socket at ½ in. Above Base, in.	
	Nominal, ft.	Limit of Minus Variation,[a] in. per ft. of length		Min.	Max.	Min.	Max.
4.....	2, 2½, 3	1/4	5/16	4 7/8	5 1/8	3 3/4	6 1/16
6.....	2, 2½, 3	1/4	3/8	7 1/16	7 1/4	8 3/16	8 5/8
8.....	2, 2½, 3	1/4	7/16	9 1/4	9 3/4	10 1/16	11
10.....	2, 2½, 3	1/4	1/2	11 3/8	12	12 3/4	13 1/4
12.....	2, 2½, 3	1/4	5/16	13 3/4	14 5/16	15 1/16	15 3/4
15.....	3, 4	1/4	5/16	17 1/16	17 11/16	18 5/16	19 1/4
18.....	3, 4	1/4	1/2	20 5/8	21 7/8	22 1/4	23
21.....	3, 4	1/4	9/16	24 1/16	25	25 7/16	26 3/4
24.....	3, 4	5/16	5/8	27 1/2	28 1/2	29 3/8	30 3/8
27.....	3, 4	5/16	5/16	31	32 1/2	33	34 1/2
30.....	3, 4	5/16	5/16	34 3/4	35 7/8	36 1/16	37 3/4
33.....	3, 4	3/8	5/8	37 5/8	38 15/16	39 7/16	41 1/4
36.....	3, 4	3/8	11/16	40 3/4	42 1/4	43 1/4	44 3/4

Size, in.	Depth of Socket, in.		Thickness of Barrel, in.		Thickness of Socket at ½ in. from Outer End, in.	
	Nominal	Min.	Nominal	Min.	Nominal	Min.
4.....	1 3/4	1 1/2	1/2	7/16	7/16	3/8
6.....	2 1/2	2	5/8	9/16	1/2	7/16
8.....	2 1/2	2 1/4	3/4	11/16	5/8	1/2
10.....	2 3/4	2 3/8	7/8	13/16	5/8	5/16
12.....	2 3/4	2 1/2	1	15/16	11/16	11/16
15.....	2 7/8	2 5/8	1 1/4	1 5/16	11/16	7/8
18.....	3	2 3/4	1 1/2	1 3/4	1 3/16	1 1/16
21.....	3 1/4	3	1 3/4	1 5/8	1 1/2	1 3/16
24.....	3 3/8	3 1/8	2	1 7/8	1 1/2	1 3/8
27.....	3 1/2	3 1/4	2 1/4	2 1/16	1 11/16	1 5/16
30.....	3 5/8	3 3/8	2 1/2	2 3/8	1 7/8	1 3/4
33.....	3 3/4	3 1/2	2 5/8	2 1/2	2	1 15/16
36.....	4	3 3/4	2 3/4	2 5/8	2 1/16	1 7/8

[a] There is no limit for plus variation.

Not more than 10 per cent of the inner surface of any pipe barrel shall be bare of glaze except the socket, where it may be entirely absent. Glazing shall not be required on the outer surface of the barrel at the spigot end for a distance from the end of the pipe equal to the specified depth of socket. There shall

be no well defined network of crazing lines or hair cracks.

Blisters

26. No blisters shall exceed 3 in. in diameter, and no blisters or pimples shall project more than $\frac{1}{8}$ in. above the surrounding surface of the pipe for sizes up to and including 18 in. in internal diameter. For sizes over 18 in. in internal diameter, no blisters shall exceed in diameter more than 2 in. per foot of the internal diameter of the pipe, nor project above the surrounding surface of the pipe more than $\frac{1}{8}$ in. per foot of the internal diameter of the pipe.

Finish of Ends

27. (*a*) The ends of the pipes shall be square with their longitudinal axis, except as provided in Table II.

(*b*) The inner surface of the socket and the outer surface of the spigot end of the pipe shall be scored by triangular-shaped or semicircular-shaped rings about $\frac{1}{8}$ in. in depth. The minimum number of scorings shall be as follows:

Size of Pipe, in.	Number of Scorings, min.
4 and 6	1
8 and 10	2
12 to 36	3

Fittings

28. (*a*) Fittings shall have a plain spigot end and a socket end corresponding in all respects with the dimensions specified for pipes of the corresponding size. The nominal laying length of T and Y branch fittings shall be as follows: 4-in. fittings, 1 ft.; 6-in. fittings, 1½ ft.; 8 to 36-in. fittings, 3 ft. Tolerances on the dimensions of fittings shall be the same as for straight pipe. All fittings shall conform in finish to the requirements for pipe prescribed in Sections 25 to 27.

(*b*) Slants shall have their spigot ends cut at an angle of approximately 60 deg. with the longitudinal axis.

(*c*) Curves shall be at angles of approximately 90, 45, and 30 deg. as required.

(*d*) Branches shall be furnished with the spur or spurs of the size or sizes specified, securely and completely fastened in the process of manufacture to the barrel of the pipe. T-branches and double T-branches shall have their axes perpendicular to the longitudinal axis of the pipe. Y-branches and double Y-branches shall have their axes approximately 60 deg. from the longitudinal axis of the pipe, measured from the socket end. All spurs shall terminate in sockets, and the barrel of the spurs shall be of sufficient length to permit making a proper joint when the connecting pipe is inserted in the spur socket.

(*e*) Channel or split pipe, curves, and branches shall be approximate half sections of the corresponding size of straight pipe and fittings.

Marking

29. Each length of pipe over 6 in. in diameter shall bear the initials or name of the person, company, or corporation by whom manufactured, and the location of the plant. The markings shall be indented on the exterior of the pipe near the socket and shall be plainly legible for purpose of identification.

INSPECTION AND REJECTION

Inspection

30. All pipe shall be subject to inspection at the factory, trench, or other point of delivery by a competent inspector employed by the purchaser. The purposes of the inspection shall be to cull and reject pipe that, independent of the physical tests herein specified, fail to conform to the requirements of these specifications.

Rejection

31. Pipe shall be subject to rejection on account of any of the following:

(*a*) Variations in any dimension exceeding the permissible variations given in Table II,

(*b*) Fractures or cracks passing through the barrel or socket, except that a single crack at the spigot end of the pipe not exceeding 75 per cent of the depth of the socket, or a single fracture in the socket not exceeding 3 in. around the circumference nor 2 in. lengthwise may be permitted,

(*c*) Chips or fractures on the interior of the pipe exceeding 2 in. in length, 1 in. in width, and of a depth more than one fourth of the thickness of the shell,

(*d*) Blisters that are broken or exceed the dimensions specified in Section 26,

(*e*) Fire cracks or hair cracks sufficient to impair the strength, durability, or serviceability of the pipe,

(*f*) Variation of more than $\frac{1}{8}$ in. per linear foot in alignment of a pipe intended to be straight,

(*g*) Glaze that does not conform to the requirements specified in Section 25, and

(*h*) Insecure attachment of spurs on branches.

Marking of Rejected Specimens

32. All pipe accepted may be plainly marked by the inspector. Rejected pipe shall not be marked or defaced so as to impair their value, but shall be replaced by the manufacturer or seller with pipe that meets the requirements of these specifications, without additional cost to the purchaser.

Tentative Specifications for

EXTRA STRENGTH CLAY PIPE[1]

A.S.T.M. Designation: C 200 – 44 T

ISSUED, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These specifications cover extra strength clay pipe intended to be used for the conveyance of sewage, industrial wastes, and storm water.

NOTE.—Attention is called to the Tentative Specifications for Standard Strength Clay Sewer Pipe (A.S.T.M. Designation: C 13)[3] and the Recommended Practice for Laying Sewer Pipe (A.S.T.M. Designation: C 12).[3]

Class

2. Pipe manufactured according to these specifications shall be known as "Extra Strength Clay Pipe."

MATERIALS AND MANUFACTURE

Materials and Manufacture

3. (*a*) Clay pipe shall be manufactured from surface clay, fire clay, or shale, or a combination of these materials.

(*b*) *Surface clay* is an unconsolidated, unstratified clay,[4] occurring on the surface.

(*c*) *Fire clay* is sedimentary clay[4] of low flux content.

NOTE.—Fire clay is usually associated with coal measures.

(*d*) *Shale* is a thinly stratified, consolidated, sedimentary clay[4] with well-marked cleavage parallel to the bedding.

(*e*) These materials or any combination thereof, when molded into pipe and subjected to suitable temperatures, shall yield a product that will be strong, durable, serviceable, free of objectionable defects, and shall conform to these specifications.

RESISTANCE TO ACTION OF ACIDS

Tests at Purchaser's Option

4. Tests for resistance to action of acids shall be optional with the purchaser, and shall be made only when specified by him in advance or in the

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-4 on Clay Pipe.

[2] Accepted by Committee E-10 on Standards, August 28, 1944.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

[4] The definitions for the terms "surface clay," "fire clay," and "shale" are based on the following definition for the term "clay":

Clay.—An earthy or stony mineral aggregate consisting essentially of hydrous silicates of alumina, plastic when sufficiently pulverized and wetted, rigid when dry, and vitreous when fired at a sufficiently high temperature.

"call for bids." The purpose of this test is to determine the resistance of pipe to the action of acids.

Reagent

5. When testing with either hydrochloric, nitric, sulfuric, or acetic acid, as specified by the purchaser, a 1 N[5] acid solution shall be used.

Test Specimens

6. (*a*) Whenever possible the test specimens shall be selected from pipe that previously have been broken in the strength test. Each specimen shall be not less than 2 by 2 in. in face area and equal in thickness to the thickness of the barrel of the pipe, and shall weigh not more than 200 g. The specimen shall be free of cracks or shattered edges.

(*b*) At least one specimen shall be taken from pipe of each size in the lot or shipment being tested and shall be marked with graphite crayon with the same designation as the pipe from which it was taken. The results shall be reported separately for each individual specimen.

Weighing Apparatus

7. The balance used in weighing the specimen shall be sensitive to 0.01 g. when loaded with 200 g.

Procedure

8. (*a*) The specimens to be tested shall be dried to constant weight at a temperature between 210 and 230 F.

(*b*) The specimens, upon reaching constant weight, shall be suspended and completely immersed in the test solution at a temperature between 70 and 90 F. for a period of 48 hr., then removed from the solution and carefully and thoroughly washed with hot water, allowing the washings to run into the solution in which the specimen was immersed. The solution shall be filtered and the filter washed with hot water, adding the washings to the filtrate. To the filtrate shall be added 5 ml. of H_2SO_4 (sp. gr. 1.84). The solution shall then be evaporated (avoiding loss by spattering) to about 5 ml., transferred to a porcelain crucible (previously ignited to constant weight), and heated cautiously to dryness. The residue shall then be ignited to constant weight.

Calculation

9. The percentage of acid-soluble matter shall be calculated as follows:

$$\text{Acid-soluble matter, per cent} = \frac{R}{W} \times 100$$

where:

R = weight of the residue in the porcelain crucible, and

W = weight of the dry specimen before immersion.

Acceptance or Rejection

10. When the test for resistance to action of acids is specified, the pipe of each size and shipment shall be acceptable only when the percentage of acid-soluble matter for specimens representing such pipe does not exceed 0.25 per cent.

PHYSICAL TEST REQUIREMENTS

Crushing Strength Tests

Test Specimens

11. (*a*) The specimens for purpose of tests shall be sound, full-size pipe and shall be selected by the purchaser or his representative at the point or points designated by him when placing the order.

(*b*) The manufacturer or seller shall furnish, without charge, specimens for tests, up to 0.5 per cent of the number of

[5] These 1 N solutions should contain, respectively, 36.5, 63, 49, and 60 g. of the acid per liter of solution. For the purpose of these tests the solution can be prepared by taking the following volumes of acid and diluting to 1 liter: H_2SO_4 (sp. gr. 1.84), 28.5 ml.; HCl (sp. gr. 1.18), 88.9 ml.; HNO_3 (sp. gr. 1.42), 65 ml.; and glacial acetic acid (sp. gr. 1.05), 57.7 ml.

pipe in each size of pipe furnished, except that in no case shall less than two specimens be furnished.

Measurement and Observation of Specimens

12. (*a*) The specimens shall first be freed from all visible moisture. When dry, each specimen shall be measured and inspected. The results of these observations shall be recorded.

(*b*) *Defective Specimens.*—Specimens that are observed to have fire cracks or other defects in form or dimensions, in excess of the limits permitted in these specifications, shall be discarded and replaced with additional specimens from the shipment.

Test Bearings

13. Pipe shall be tested for crushing strength by either the three-edge-bearing method or by the sand-bearing method as described in Sections 16 and 17. Either of the two specified bearing methods shall be used on retests as provided for in Section 22 (*b*) and (*c*).

Apparatus and Application of Load

14. (*a*) Any prime mover or hand power device that will apply the load at a uniform rate of about 2000 lb. per min., or in increments of not more than 100 lb. at the same rate, may be used in making the test.

(*b*) The center of the load shall be applied to the top bearing block at a point distant from the spigot end of the pipe equal to one half of the over-all length of the pipe. The test load shall be applied to the upper bearing block in such a way as to leave the bearing free to move in a vertical plane passing midway between the lower bearings. In testing pipe that is "out of straight," the lines of the bearings chosen shall be from those which appear to give most favorable conditions for fair test.

(*c*) The pipe shall be allowed to stand under load no longer than is required to apply the load and to observe and record it.

(*d*) The testing machine shall be substantial and rigid throughout, so that the distribution of the load will not be affected appreciably by the deformation or yielding of any part. The bearings shall be sufficiently rigid to transmit and receive uniform loads throughout their lengths without deflection, and shall be so attached to the machine as to transmit and receive the maximum loads produced by the tests without lost motion, vibration, or sudden shock.

(*e*) The bearings and the specimen shall be accurately centered so as to secure a symmetrical distribution of the loading on each side of the center of the pipe in every direction.

(*f*) The load shall be applied until the pipe yields by cracks passing through the shell. At the discretion of the inspector the loading may be stopped before rupture of the pipe, provided the load is at least equal to the required average strength of the pipe.

Calculation and Report

15. The crushing strength shall be calculated by dividing the breaking load or applied load as provided in Section 14 (*f*) by the net inside length of the barrel of the pipe, measuring from the bottom of the socket to the end of the spigot. The individual results of the tests of pipe of each size or each shipment shall be tabulated separately so as to show the percentage that fails to conform to the requirements of each test.

Three-Edge-Bearing Method

16. When the three-edge-bearing method is used, the ends of each specimen of pipe shall be accurately marked in halves of the circumference prior to the test. The lower bearings shall con-

sist of two wooden strips with vertical sides having their interior top corners rounded to a radius of approximately ½ in. The strips shall be straight and shall be securely fastened to a rigid block at least 6 by 6 in. in cross-section. The interior vertical sides of the strips shall be parallel and spaced a distance apart of 1 in. per foot of pipe diameter, but in no case less than 1 in. Before the pipe is placed, a fillet of plaster of Paris or other equalizing material, such as sand and sulfur, thick enough to compensate for the inequalities of the pipe barrel shall be cast on and between the lower bearings. The pipe shall be placed on the fillet while the plaster of Paris is still somewhat plastic. The upper bearing shall be a rigid wooden block at least 6 by 6 in. in cross-section, straight and true from end to end. A fillet of plaster of Paris thick enough to compensate for the inequalities of the pipe barrel shall also be cast along the length of the crown. The upper bearing shall be brought in contact while the plaster of Paris is still somewhat plastic. The upper and lower bearings shall extend the full length of the pipe exclusive of the socket.

Sand-Bearing Method

17. (*a*) When the sand-bearing method is used, the ends of each specimen of pipe shall be accurately marked in quarters of the circumference prior to the test. Specimens shall be carefully bedded in sand, above and below, for one fourth the circumference of the pipe measured on the middle line of the barrel. The depth of the bedding above and below the pipe at the thinnest points shall be one half the radius of the middle line of the barrel.

(*b*) The sand used shall be clean and moist and shall be such as will pass a 4760-micron (No. 4) sieve conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11).[3] The sand in the lower bearing shall be loose when the pipe is placed.

(*c*) The top bearing frame shall not be allowed to come in contact with the pipe nor with the top bearing plate. The upper surface of the sand in the top bearing shall be struck off level with a straightedge and shall be covered with a rigid top bearing plate, the lower surface of which is a true plane, made of heavy timbers or other rigid material, capable of distributing the test load uniformly without appreciable bending. The test load shall be applied at the exact center of this top bearing plate, in such a manner as to permit free motion of the plate in all directions. For this purpose a spherical bearing is preferred, but two rollers at right angles may be used.

(*d*) The test may be made without the use of a testing machine, by piling weights directly on a platform resting on the top bearing plate; provided, however, that the weights be piled symmetrically about a vertical line through the center of the pipe, and that the platform shall not be allowed to touch the top bearing frame.

(*e*) The frames of the top and bottom bearings shall be made of timbers so heavy as to avoid appreciable bending by the side pressure of the sand. The interior surfaces of the frames shall be dressed. No frame shall come in contact with the pipe during the test. A strip of cloth may be attached, if desired, to the inside of the upper frame on each side, along the lower edge, to prevent the escape of sand between the frame and the pipe.

Absorption Test

Test Specimens

18. (*a*) The test specimens shall be sound pieces, of the full thickness of the

barrel of the pipe, with all edges broken, and may be from pipe broken in the strength tests. They shall be from 12 to 20 sq. in. in area, and shall be as nearly square as they can be readily prepared. They shall be free of observable cracks or shattered edges, and shall contain laminations and fissures only to the extent that these are representative of the pipe from which the specimens were taken.

(*b*) Each specimen shall be marked so that it may be identified with the pipe used in the strength test from which the specimen was taken. The markings shall be applied so that the pigment used shall cover not more than 1 per cent of the total superficial area of the specimen.

(*c*) One specimen shall be tested from each pipe broken in the strength test.

Weighing Apparatus

19. The balance used shall be sensitive to 0.5 g. when loaded with 1 kg., and weighings shall be read at least to the nearest gram. When other than metric weights are used, the same degree of accuracy shall be obtained.

Procedure

20. (*a*) The specimens shall be dried to constant weight in a drier or oven at a temperature of not less than 110 C. (230 F.).

(*b*) The dried specimens shall be placed in a suitable wire receptacle, packed tightly enough to prevent jostling, covered with distilled water or rain water, raised to the boiling point and boiled for 5 hr., and then cooled in water to a final temperature of 15 to 25 C. (59 to 77 F.). When cool, the specimens shall be removed from the water and allowed to drain for not more than 1 min. The superficial moisture shall then be removed by a towel or blotting paper, and the specimens immediately weighed.

Calculation and Report

21. The absorption of each specimen shall be calculated as percentage of the initial dry weight. The results shall be reported separately for each individual specimen, and as averages for the pipe of each size and for the shipment.

ACCEPTANCE OR REJECTION ON RESULTS OF TESTS

Crushing Strength and Absorption

22. (*a*) The crushing strength shall conform to the requirements prescribed in Table I and the absorption shall not exceed 8 per cent, except as provided in Paragraphs (*b*), (*c*), and (*d*).

TABLE I.—CRUSHING STRENGTH REQUIREMENTS FOR EXTRA-STRENGTH CLAY SEWER PIPE.

Nominal Size, in.	Average Strength, min., lb. per linear ft.	
	Three-Edge-Bearing Method	Sand-Bearing Method
6.....	2000	2850
8.....	2000	2850
10.....	2000	2850
12.....	2250	3200
15.....	2750	3925
18.....	3300	4700
21.....	3850	5500
24.....	4400	6300
30.....	5000	7100
36.....	6000	8575

(*b*) Failure of more than 20 per cent of the specimens of a size to conform to the requirements of the strength tests or the absorption tests or both shall result in rejection of all the pipe of that size in the shipment or delivery; except that in the event of 20 per cent of the specimens in any size failing to meet the requirements, the manufacturer or seller may, with the consent of the purchaser, furnish for test, without charge, additional specimens from the same shipment to be selected as specified in Section 11. In case more than 80 per cent of the speci-

mens tested, including those first tested, shall show substantial conformance for each of the various tests performed, then the entire shipment or delivery for this size shall be accepted; otherwise it shall be rejected.

(*c*) However, in case of failure of pipe to conform to the strength requirements under the specified method, and the accuracy of the testing machine is questioned, a retest at the request of the manufacturer may be made upon a machine whose accuracy is known, or the machine originally used shall be recalibrated before the retest is made.

(*d*) In addition to the requirements in Paragraph (*b*), failure of 10 per cent or more of the individual specimens tested to develop 75 per cent of the average crushing strength requirements shall be cause for the rejection of the shipment, but the manufacturer or seller may cull the pipe and submit the balance of the shipment for retest, and if the shipment then conforms to all of the requirements of these specifications it shall be accepted.

SIZES AND DIMENSIONS, WORKMANSHIP AND FINISH

Sizes and Dimensions

23. Pipe shall be furnished of the sizes and of the dimensions, including permissible variations, prescribed in Table II. When more than one length is permitted, the purchaser shall indicate, at the time of purchase, which lengths shall be furnished; unless so indicated, the manufacturer shall furnish such lengths as he may elect.

Straightness

24. Pipe intended to be straight shall have a maximum ordinate as measured from the concave side of the pipe not to exceed $\frac{1}{8}$ in. per foot of length.

Salt Glaze[6]

25. The glaze shall consist of a continuous layer of salt glaze substantially free of large blisters or large pimples. Not more than 10 per cent of the inner surface of any pipe barrel shall be bare of glaze except the socket, where it may be entirely absent. Glazing shall not be required on the outer surface of the barrel at the spigot end for a distance from the end of the pipe equal to the specified depth of socket. There shall

TABLE II.—DIMENSIONS OF EXTRA-STRENGTH CLAY SEWER PIPE.

Nominal Size, in.	Laying Length			Outside Diameter of Barrel,[b] in.		Inside Diameter of Socket at ½ in. Above Base, in.	
	Nominal, ft.	Limit of Minus Variation,[a] in. per ft. of length	Maximum Difference in Length of Two Opposite Sides, in.	Min.	Max.	Min.	Max.
6	2, 2½, 3	1/4	3/8	7 1/8	7 3/8	8 3/8	8 5/8
8	3	1/4	1/8	9 1/4	9 3/4	10 1/2	11
10	3	1/4	1/16	11 1/8	12	12 3/4	13 1/4
12	3	1/4	1/16	13 3/4	14 3/8	15 1/8	15 3/4
15	3, 4	1/4	1/16	17 1/16	17 15/16	18 1/8	19 1/4
18	3, 4	1/4	1/16	20 1/8	21 1/16	22 1/4	23
21	3, 4	1/4	1/16	24 1/8	25	25 7/8	26 3/4
24	3, 4	3/8	1/16	27 1/4	28 1/4	29 3/8	30 3/8
30	3, 4	3/8	5/16	34 3/16	35 5/16	36 1/2	37 3/4
36	3, 4	3/8	11/16	40 3/4	42 1/4	43 1/2	44 3/4

Nominal Size, in.	Depth of Socket, in.		Thickness of Barrel, in.		Thickness of Socket at ½ in. from Outer End, in.	
	Nominal	Min.	Nominal	Min.	Nominal	Min.
6	2 1/4	2	11/16	9/16	1/2	7/16
8	2 1/2	2 1/4	7/8	3/4	9/16	1/2
10	2 5/8	2 3/8	1	7/8	5/8	7/16
12	2 3/4	2 1/2	1 1/16	1 1/16	3/4	11/16
15	2 7/8	2 5/8	1 1/8	1 3/8	15/16	7/8
18	3	2 3/4	1 7/16	1 3/4	1 1/16	1 1/16
21	3 1/4	3	2 1/4	2	1 3/16	1 1/16
24	3 3/8	3 1/4	2 1/2	2 1/4	1 1/2	1 3/8
30	3 5/8	3 3/4	3	2 3/4	1 7/8	1 3/4
36	4	3 3/4	3 1/2	3 1/4	2 1/16	1 7/8

[a] There is no limit for plus variation.
[b] The average actual inside diameters of pipe having the nominal thickness of barrel shown in Table II may be smaller than the nominal sizes.

[6] Unglazed pipe cannot be demanded by the purchaser but may be accepted at his discretion.

be no well defined network of crazing lines or hair cracks.

Blisters

26. No blisters shall exceed 3 in. in diameter and no blisters or pimples shall project more than $\frac{1}{8}$ in. above the surrounding surface of the pipe for sizes up to and including 18 in. in internal diameter. For sizes over 18 in. in internal diameter no blisters shall exceed in diameter more than 2 in. per foot of the internal diameter of the pipe, nor project above the surrounding surface of the pipe more than $\frac{1}{8}$ in. per foot of the internal diameter of the pipe.

Finish of Ends

27. (*a*) The ends of the pipe shall be square with their longitudinal axis, except as provided in Table II.

(*b*) The inner surface of the socket and the outer surface of the spigot end of the pipe shall be scored by triangular-shaped or semicircular-shaped rings about $\frac{1}{8}$ in. in depth. The minimum number of scorings shall be as follows:

Size of Pipe, in.	Number of Scorings, min.
6	1
8 and 10	2
12 to 21	3
24 and 30	4
36	5

Fittings

28. (*a*) All T and Y branches shall terminate in sockets, and the distance from the spigot end of branch fittings to the junction of a spur shall be sufficient to permit acceptable jointing to adjacent pipe. The nominal laying length of T and Y branch fittings shall be as follows: 6-in. fittings, $1\frac{1}{2}$ ft.; 8 to 36-in. fittings, 3 ft. Tolerances on the dimensions of fittings shall be the same as for straight pipe. All fittings shall conform in finish to the requirements for pipe prescribed in Sections 25 to 27.

(*b*) Slants shall have their spigot ends cut at an angle of approximately 60 deg. with the longitudinal axis.

(*c*) Curves shall be at angles of approximately 90, 45, and 30 deg. as required.

(*d*) Branches shall be furnished with the spur or spurs of the size or sizes specified, securely and completely fastened in the process of manufacture to the barrel of the pipe. T-branches and double T-branches shall have their axes perpendicular to the longitudinal axis of the pipe. Y-branches and double Y-branches shall have their axes approximately 60 deg. from the longitudinal axis of the pipe, measured from the socket end. All spurs shall terminate in sockets, and the barrel of the spurs shall be of sufficient length to permit making a proper joint when the connecting pipe is inserted in the spur socket.

(*e*) Channel or split pipe, curves, and branches shall be approximate half sections of the corresponding size of straight pipe and fittings.

Marking

29. Each length of pipe shall bear the initials or name of the person, company, or corporation by whom manufactured, the location of the plant, and the symbol "ES." The markings shall be indented on the exterior of the pipe near the socket and shall be plainly legible for purpose of identification.

INSPECTION AND REJECTION

Inspection

30. All pipe shall be subject to inspection at the factory, trench, or other point of delivery by a competent inspector employed by the purchaser. The purposes of the inspection shall be to cull and reject pipe that, independent of the physical tests herein specified, fail to conform to the requirements of these specifications.

Rejection

31. Pipe shall be subject to rejection on account of any of the following:

(*a*) Variations in any dimension exceeding the permissible variations given in Table II,

(*b*) Fractures or cracks passing through the barrel or socket, except that a single crack at the spigot end of the pipe not exceeding 75 per cent of the depth of the socket, or a single fracture in the socket not exceeding 3 in. around the circumference nor 2 in. lengthwise may be permitted,

(*c*) Chips or fractures on the interior of the pipe exceeding 2 in. in length, 1 in. in width, and of a depth more than one fourth of the thickness of the shell,

(*d*) Blisters that are broken or exceed the dimensions specified in Section 26,

(*e*) Fire cracks or hair cracks sufficient to impair the strength, durability, or serviceability of the pipe,

(*f*) Variation of more than $\frac{1}{8}$ in. per linear foot in alignment of a pipe intended to be straight,

(*g*) Glaze that does not conform to the requirements specified in Section 25, and

(*h*) Insecure attachment of spurs on branches.

Marking of Rejected Specimens

32. All pipe accepted may be plainly marked by the inspector. Rejected pipe shall not be marked so as to be defaced or to impair their value, but shall be replaced by the manufacturer or seller with pipe that meets the requirements of these specifications, without additional cost to the purchaser.

Tentative Method of Test for

SOUNDNESS OF AGGREGATES BY USE OF SODIUM SULFATE OR MAGNESIUM SULFATE[1]

A.S.T.M. Designation: C 88 – 44 T

ISSUED, 1931; REVISED, 1932, 1935, 1937, 1939, 1941, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. This method covers the procedure to be followed in testing aggregates to determine their resistance to disintegration by saturated solutions of sodium sulfate or magnesium sulfate. It furnishes information helpful in judging the soundness of aggregates subject to weathering action, particularly when adequate information is not available from service records of the material exposed to actual weathering conditions. Attention is called to the fact that test results by the use of the two salts differ considerably and care must be exercised in fixing proper limits in any specifications which may include requirements for these tests.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Sieves.*—Sieves with square openings of the following sizes conforming to the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials,[3] for sieving the samples in accordance with Sections 4 and 5:

Fine Series	Coarse Series
No. 100 (149 micron)	$\frac{3}{8}$ in.
No. 50 (297 micron)	$\frac{3}{4}$ in.
No. 30 (590 micron)	$1\frac{1}{2}$ in.
No. 16 (1190 micron)	$2\frac{1}{2}$ in.
No. 8 (2380 micron)	larger sizes by
No. 4 (4760 micron)	1-in. spread.

(*b*) *Containers.*—Containers for immersing the samples of aggregate in the solution, in accordance with the procedure described in this method, shall be perforated in such a manner as to permit of free access of the solution to the sample and of drainage of the solution from the sample without loss of aggregate. The volume of the solution

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates.

[2] Latest revision accepted by the Society at annual meeting, June, 1944.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

in which samples are immersed shall be at least five times the volume of the sample immersed at any one time.

NOTE.—Baskets made of suitable wire mesh or sieves with suitable openings are satisfactory containers for the samples.

(*c*) *Temperature Regulation.*—Suitable means for regulating the temperature of the samples during immersion in the sodium sulfate or magnesium sulfate solution shall be provided.

(*d*) *Balances.*—For weighing fine aggregate, a balance having a capacity of not less than 500 g., sensitive to at least 0.1 g., shall be used; for weighing coarse aggregate, a balance having a capacity of not less than 5000 g., sensitive to at least 1 g., shall be used.

(*e*) *Drying Oven.*—The drying oven shall provide a free circulation of air through the oven and shall be capable of maintaining a temperature of 105 to 110 C. (221 to 230 F.).

Special Solutions Required

3. (*a*) *Sodium Sulfate Solution.*—The saturated solution of sodium sulfate shall be prepared by dissolving a c.p., U.S.P., or equal grade of the salt in water at a temperature of 25 to 30 C. (77 to 86 F.). Sufficient salt (Note), of either the anhydrous (Na_2SO_4) o the crystalline ($Na_2SO_4 \cdot 10H_2O$) form,[4] shall be added to insure not only saturation but also the presence of excess crystals when the solution is ready for use in the tests. The mixture shall be thoroughly stirred during the addition of the salt and the solution shall be stirred at frequent intervals until used. The solution shall be cooled to a temperature of 21 ± 1 C. (70 ± 2 F.) and maintained at that temperature for at least 48 hr. before use; it shall be thoroughly stirred immediately before use and, when used, shall have a specific gravity of not less than 1.151 and not greater than 1.174.

NOTE.—For the solution, 215 g. of anhydrous salt or 700 g. of the decahydrate per liter of water are sufficient for saturation at 22 C. However, since these salts are not completely stable and since it is desirable that an excess of crystals be present, the use of not less than 350 g. of the anhydrous salt or 750 g. of the decahydrate salt per liter of water is recommended.

(*b*) *Magnesium Sulfate Solution.*—The saturated solution of magnesium sulfate shall be made by dissolving a c.p., U.S.P., or equal grade of the salt in water at a temperature of 25 to 30 C. (77 to 86 F.). Sufficient salt (Note), of either the anhydrous ($MgSO_4$) or the crystalline ($MgSO_4 \cdot 7H_2O$) (Epsom Salt) form, shall be added to insure saturation and the presence of excess crystals when the solution is ready for use in the tests. The mixture shall be thoroughly stirred during the addition of the salt and the solution shall be stirred at frequent intervals until used. The solution shall be cooled to a temperature of 21 ± 1 C. (70 ± 2 F.) and maintained at that temperature for at least 48 hr. before use; it shall be thoroughly stirred immediately before use and, when used, shall have a specific gravity of not less than 1.295 and not more than 1.308.

NOTE.—For the solution, 350 g. of anhydrous salt or 1230 g. of the heptahydrate per liter of water are sufficient for saturation at 23 C. However, since these salts are not completely stable, with the hydrous salt being the more stable of the two, and since it is desirable that an excess of crystals be present, it is recommended that the heptahydrate salt be used and in an amount of not less than 1400 g. per liter of water.

Samples

4. (*a*) *Fine Aggregate.*—Fine aggregate for the test shall be passed through

[4] Experience with the test method indicates that a grade of sodium sulfate designated by the trade as dried powder, which may be considered as approximately anhydrous, is the most practical for use. That grade is more economically available than the anhydrous form. The decahydrate sodium sulfate presents difficulties in compounding the required solution on account of its cooling effect on the solution.

a $\frac{3}{8}$-in. sieve. The sample shall be of such size that it will yield not less than 100 g. of each of the following sizes, which shall be available in amounts of 5 per cent or more, expressed in terms of the following sieves:

Retained on Sieve	Passing Sieve
No. 50 (297 micron)	No. 30 (590 micron)
No. 30 (590 micron)	No. 16 (1190 micron)
No. 16 (1190 micron)	No. 8 (2380 micron)
No. 8 (2380 micron)	No. 4 (4760 micron)
No. 4 (4760 micron)	$\frac{3}{8}$ in.

(*b*) *Coarse Aggregate.*—Coarse aggregate for the test shall consist of material from which the sizes finer than the No. 4 (4760-micron) sieve have been removed. The sample shall be of such a size that it will yield not less than the following amounts of the different sizes, which shall be available in amounts of 5 per cent or more:

Size (Square-Opening Sieves)	
No. 4 to $\frac{3}{8}$ in.	300 g.
$\frac{3}{8}$ to $\frac{3}{4}$ in.	1000 g.
Consisting of:	
$\frac{3}{8}$ to $\frac{1}{2}$-in. material	33 per cent
$\frac{1}{2}$ to $\frac{3}{4}$-in. material	67 per cent
$\frac{3}{4}$ to 1$\frac{1}{2}$ in.	1500 g.
Consisting of:	
$\frac{3}{4}$ to 1-in. material	33 per cent
1 to 1$\frac{1}{2}$-in. material	67 per cent
1$\frac{1}{2}$ to 2$\frac{1}{2}$-in.	3000 g.
Consisting of:	
1$\frac{1}{2}$ to 2-in. material	50 per cent
2 to 2$\frac{1}{2}$-in. material	50 per cent
Larger sizes by 1-in. spread in sieve size, each fraction	3000 g.

(*c*) Should the samples contain less than 5 per cent of any of the sizes specified in Paragraph (*a*) or (*b*), that size shall not be tested, but, for the purpose of calculating the test results, it shall be considered to have the same loss in sodium sulfate or magnesium sulfate treatment as the average of the next smaller and the next larger size, or if one of these sizes is absent, it shall be considered to have the same loss as the next larger or next smaller size, whichever is present.

Preparation of Test Sample

5. (*a*) *Fine Aggregate.*—The sample of fine aggregate shall be thoroughly washed on a No. 50 (297-micron) sieve, dried to constant weight at 105 to 110 C. (221 to 230 F.), and separated into the different sizes by sieving, as follows: Make a rough separation of the graded sample by means of a nest of the standard sieves specified in Section 4 (*a*). From the fractions obtained in this manner select samples of sufficient size to yield 100 g. after sieving to refusal. (In general, a 110-g. sample will be sufficient.) Fine aggregate sticking in the meshes of the sieves shall not be used in preparing the samples. Samples consisting of 100 g. shall be weighed out of each of the separated fractions after final sieving and placed in separate containers for the test.

(*b*) *Coarse Aggregate.*—The sample of coarse aggregate shall be thoroughly washed and dried to constant weight at 105 to 110 C. (221 to 230 F.) and shall be separated into the different sizes shown in Section 4 (*b*) by sieving to refusal. The proper weight of sample for each fraction shall be weighed out and placed in separate containers for the test. In the case of fractions coarser than the $\frac{3}{4}$-in. sieve, the number of particles shall be counted.

(*c*) *Ledge Rock.*—For testing ledge rock, the sample shall be prepared by breaking it into fragments reasonably uniform in size and shape and weighing approximately 100 g. each. The test sample shall weigh 5000 g. $\pm$ 2 per cent. The sample shall be thoroughly washed and dried previous to test as described in Paragraph (*b*).

Procedure

6. (*a*) *Storage of Samples in Solution.*—The samples shall be immersed in the prepared solution of sodium sulfate or magnesium sulfate for not less than 16

hr. nor more than 18 hr. in such a manner that the solution covers them to a depth of at least ½ in. The containers shall be covered to reduce evaporation and prevent the accidental addition of extraneous substances. The samples immersed in the solution shall be maintained at a temperature of 21 ± 1 C. (70 ± 2 F.) for the immersion period.

(*b*) *Drying Samples After Immersion.*—After the immersion period the aggregate sample shall be removed from the solution, permitted to drain, and placed in the drying oven. The temperature of the oven shall have been brought previously to 105 to 110 C. (221 to 230 F.). Care shall be exercised to avoid loss of any of the aggregate particles or, in the case of fine aggregate, of any detritus coarser than a No. 100 (149-micron) sieve (Note). The samples shall be dried to constant weight at the specified temperature. After drying, the samples shall be allowed to cool to room temperature, when they shall again be immersed in the prepared solution as described in Paragraph (*a*).

NOTE.—In the case of coarse aggregate the detritus should also be saved if the complete analysis suggested in the Note in Section 7 (*b*) is made.

(*c*) *Number of Cycles.*—The process of alternate immersion and drying shall be repeated until the required number of cycles is obtained.

Quantitative Examination

7. The quantitative examination (Note) shall be made as follows:

(*a*) After the completion of the final cycle and after the sample has cooled, the sample shall be washed free from the sodium sulfate or magnesium sulfate as determined by the reaction of the wash water with barium chloride ($BaCl_2$).

(*b*) After the sodium sulfate or magnesium sulfate solution has been removed, each fraction of the sample shall be dried to constant weight at 105 to 110 C. (221 to 230 F.), weighed, and, except in the case of ledge rock, sieved over the same sieve on which it was retained before the test. The particles retained on this sieve shall be weighed and the weight recorded.

NOTE.—In addition to the procedure described in Paragraphs (*a*) and (*b*), it is suggested that additional information of value will be obtained by examining each fraction visually in order to determine whether there is any evidence of excessive splitting of the grains. It is suggested also that additional information of value will be obtained if, after treating each separate fraction of the sample as described in Paragraph (*b*), all sizes, including detritus, are combined and a sieve analysis made using a complete set of sieves for the determination of the fineness modulus. The results of the sieve analysis shall be recorded as cumulative percentages retained on each sieve.

(*c*) *Alternative Procedure.*—After the sodium sulfate or magnesium sulfate solution has been removed, each fraction of the sample shall be dried to constant weight at 105 to 110 C. (221 to 230 F.), weighed, and, except in the case of ledge rock, sieved over a sieve having square openings one half the size of the sieve on which the material was originally retained. The particles retained on this sieve shall be weighed and the weight recorded.

(*d*) In the case of ledge rock the loss in weight shall be determined by subtracting from the original weight of the sample the final weight of all fragments which have not broken into three or more pieces.

Qualitative Examination

8. (*a*) Fractions of samples coarser than ¾ in. shall be examined qualitatively after each immersion and quantitatively at the completion of the test.

(*b*) The qualitative examination and record shall consist of two parts: (*1*)

observing the effect of the action (Note) by the sodium sulfate or magnesium sulfate solution and the nature of the action, and (*2*) counting the number of particles affected.

NOTE.—Many types of action may be expected. In general, they may be classified as disintegration, splitting, crumbling, cracking, flaking, etc.

While only particles larger than ¾ in. in size are required to be examined qualitatively, it is recommended that examination of the smaller sizes be made in order to determine whether there is any evidence of excessive splitting.

received for examination or, preferably, on the average grading of the material from that portion of the supply of which the sample is representative. In these calculations sizes finer than the No. 50 sieve shall be assumed to have 0 per cent loss.

(*d*) In the case of particles coarser than ¾ in. before test: (*1*) The number of particles in each fraction before test, and (*2*) the number of particles affected, classified as to number disintegrating,

TABLE I.—SUGGESTED FORM FOR RECORDING TEST DATA (WITH ILLUSTRATIVE TEST VALUES).

Sieve Size		Grading of Original Sample, per cent	Weight of Test Fractions Before Test, g.	Percentage Passing Finer Sieve After Test (Actual Percentage Loss)	Weighted Average (Corrected Percentage Loss)
Passing	Retained on				
SOUNDNESS TEST OF FINE AGGREGATE					
No. 100 (149 micron)		5.0	....	....	....
No. 50 (297 micron)	No. 100 (149 micron)	11.4	....	....	....
No. 30 (590 micron)	No. 50 (297 micron)	26.0	100	4.2	1.09
No. 16 (1190 micron)	No. 30 (590 micron)	25.2	100	4.8	1.21
No. 8 (2380 micron)	No. 16 (1190 micron)	17.0	100	8.0	1.36
No. 4 (4760 micron)	No. 8 (2380 micron)	10.8	100	11.2	1.21
⅜ in.	No. 4 (4760 micron)	4.6	....	11.2[a]	0.52
Totals		100.0	400	....	5.39
SOUNDNESS TEST OF COARSE AGGREGATE					
2½ in.	1½ in.	20.0	3000[b]	4.8	0.96
1½ in.	¾ in.	45.0	1500[b]	8.0	3.60
¾ in.	⅜ in.	23.0	1000[b]	9.6	2.20
⅜ in.	No. 4 (4760 micron)	12.0	300[b]	11.2	1.34
Totals		100.0	5800	....	8.10

[a] The percentage loss (11.2 per cent) of the next smaller size is used as the percentage loss for this size, since this size contains less than 5 per cent of the original sample as received. See Section 4 (*c*).
[b] Minimum amounts; larger samples may be used.

Report

9. The report shall include the following data (Note):

(*a*) Weight of each fraction of each sample before test.

(*b*) Except in the case of ledge rock, material from each fraction of the sample finer than the sieve on which the fraction was retained before test expressed as a percentage by weight of the fraction.

(*c*) Weighted average calculated from the percentage of loss for each fraction, based on the grading of the sample as splitting, crumbling, cracking, flaking, etc.

(*e*) In the case of ledge rock: (*1*) The percentage of loss calculated as described in Section 7 (*d*) and (*2*) the number of pieces affected, classified as to number disintegrating, splitting, crumbling, cracking, flaking, etc.

(*f*) Character of solution (sodium or magnesium sulfate).

NOTE.—Table I, shown with test values inserted for purpose of illustration, is a suggested form for recording test data. The test values shown might be appropriate for either salt, depending on the quality of the aggregate.

Tentative Method of Test for
SURFACE MOISTURE IN FINE AGGREGATE[1]

A.S.T.M. Designation: C 70 – 44 T

ISSUED, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. This method of test covers a procedure for determining, in the field, the amount of surface moisture in fine aggregate by displacement in water. The accuracy of the method depends upon accurate information on the bulk specific gravity of the material in a saturated surface-dried condition. The same procedure, with appropriate changes in the size of sample and dimensions of the container, may be applied to coarse aggregate.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Balance.*—A balance having a capacity of 2 kg. or more and sensitive to 0.5 g. or less.

(*b*) *Flask.*—A suitable container or flask, preferably of glass or noncorrosive metal. The container may be a pycnometer, a volumetric flask, a graduated volumetric flask or other suitable measuring device. The volume of the container shall be from 2 to 3 times the loose volume of the sample. The container shall be so designed that it can be filled to mark, or the volume of its contents read, within 0.5 ml. or less.

Sample

3. A representative sample of the fine aggregate to be tested for surface moisture content shall be selected. It shall weigh not less than 200 g. Larger samples will yield more accurate results.

Procedure

4. (*a*) The surface water content may be determined either by weight or volume. In each case the test shall be made at a temperature range of 65 to 85 F.

(*b*) *Determination by Weight.*—The container shall be filled to mark with water and the weight in grams determined. The container shall then be emptied. Enough water shall then be placed in the container to cover the sample, after which the sample of fine ag-

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates.

[2] Prior to its present publication as tentative, this method was published as tentative from 1927 to 1930, being revised in 1928 and 1930. It was adopted in 1930, published as standard from 1930 to 1944, but was withdrawn, revised, and republished as tentative in 1944.

gregate shall be introduced into the container and the entrained air removed. The container shall then be filled to the original mark and the weight in grams determined. The amount of water displaced by the sample shall be calculated as follows:

$$V_s = W_c + W_s - W$$

where:

V_s = weight of water displaced by sample, in grams,
W_c = weight of container filled to mark with water, in grams,
W_s = weight of sample, in grams, and
W = weight of container and sample, filled to mark with water, in grams.

(*c*) *Determination by Volume.*—A volume of water sufficient to cover the sample shall be measured in milliliters and placed in the container. The weighed sample of fine aggregate shall then be admitted into the container and the entrained air removed. The combined volume of the sample and the water shall be determined by direct reading when a graduated flask is used. When a pycnometer or volumetric flask of known volume is used, the combined volume of the sample and the water shall be determined by filling to the mark with a measured volume of water and subtracting this volume from the volume of the container. The amount of water displaced by the sample shall be calculated as follows:

$$V_s = V_2 - V$$

where:

V_s = volume of water displaced by sample, in milliliters,
V_2 = combined volume of sample and water, in milliliters, and
V_1 = volume of water required to cover sample, in milliliters.

Calculation

5. The percentage of surface moisture in terms of the saturated surface-dry fine aggregate and in terms of the weight of wet fine aggregate shall be calculated as follows:[3]

$$P_1 = \frac{V_s - V_d}{W_s - V_s} \times 100$$

$$P_2 = \frac{V_s - V_d}{W_s - V_d} \times 100$$

where:

P_1 = surface moisture in terms of saturated surface-dry fine aggregate, in per cent,
P_2 = surface moisture in terms of the weight of wet fine aggregate, in per cent,
V_d = the weight of the sample (W_s in Section 4 (*b*)) divided by the bulk specific gravity of the fine aggregate in a saturated surface-dried condition, determined as prescribed in the Standard Method of Test for Specific Gravity and Absorption of Fine Aggregate (A.S.T.M. Designation: C 128),[4]
V_s = weight of water displaced, in grams, and
W_s = weight of sample, in grams.

[3] These formulas are readily derived from basic relationships. For convenience express P_1 in terms of the ratio r, that is, the ratio of the weight of surface moisture to the weight of the saturated, surface-dry sample. It follows that:

$$r = \frac{W_s - \frac{W_s}{1+r}}{\frac{W_s}{1+r}} \quad \text{.....(1)}$$

If G is bulk specific gravity of the saturated-surface-dry fine aggregate, then

$$V_s = \frac{W_s}{G(1+r)} + \left(W_s - \frac{W_s}{1+r}\right) \quad \text{.....(2)}$$

where the first term gives the water displaced by the saturated-surface-dry fine aggregate and the second that displaced by the surface moisture.

From Eq. 2:

$$\frac{W_s}{1+r} = \frac{V_s - W_s}{\frac{1}{G} - 1} \quad \text{.....(3)}$$

By definition,

$$W_s = V_d\, G \quad \text{.....(4)}$$

Substituting for $\frac{W_s}{1+r}$ and W_s in Eq. 1 and simplifying,

$$r = \frac{V_s - V_d}{W_s - V_s} \quad \text{.....(5)}$$

The formula for P_2 may be derived by similar reasoning, or directly from that for P_1 since,

$$P_2 = \frac{\frac{V_s - V_d}{W_s - V_s}}{1 + \frac{V_s - V_d}{W_s - V_s}} \times 100 \quad \text{.....(6)}$$

[4] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Tentative Definition of

THE TERM AGGREGATE[1]

A.S.T.M. Designation: C 58 – 28 T

ISSUED, 1926; REVISED, 1928.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Aggregate.—In the case of materials of construction, designated inert material which when bound together into a conglomerated mass by a matrix forms concretes, mastic, mortar, plaster, etc.

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[1] Under the standardization procedure of the Society, this definition is under the jurisdiction of the A.S.T.M. Committee E-8 on Nomenclature and Definitions.

[2] This tentative definition when adopted as standard will be added to the Standard Definition of the Term Sand (A.S.T.M. Designation: C 58 – 28), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Tentative Method of Test for

AIR CONTENT (VOLUMETRIC) OF FRESHLY MIXED CONCRETE[1]

A.S.T.M. Designation: C 173 - 42T

ISSUED, 1942.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. This method of test covers the procedure for determining the quantity of air entrained in freshly mixed concrete or mortar.

NOTE.—This method is believed to be adequate for all ordinary types of concrete and mortar, except that some types of masonry mortar, particularly those containing soap-forming materials, may not permit air determinations to be made with the same degree of accuracy that may be expected in tests of concrete.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Pycnometer.*—A pycnometer, or weighing vessel, preferably cylindrical in shape and having a height between two and three times the diameter. The capacity shall be between 3 and 4 liters (Note). The pycnometer shall be fitted with a flat, watertight cover and a suitable quick-acting clamp for holding it firmly in place. A flat piece of plate glass at least $\frac{3}{8}$ in. in thickness, having a width somewhat less and a length somewhat greater than the diameter of the pycnometer, and having a sharp gaging point about 1 in. in length attached to the center of one face, shall be provided for gaging the volume of the pycnometer at a definite height above the bottom. For accuracy of this measurement the top edge and the outside surface of the bottom of the pycnometer shall be machined to true planes perpendicular to the axis of the cylinder.

NOTE.—For concrete containing aggregates of maximum size greater than $1\frac{1}{2}$ in., a larger size pycnometer is desirable. A pycnometer of 2 liters capacity should be satisfactory for mortar.

(*b*) *Balance.*—A solution balance, or one of the suspended pan type, sensitive to 0.5 g. and having a capacity of 10 kg., or more, as may be required.

(*c*) *Weights.*—Calibrated weights for the balance, such that any combination of weights in the desired range will be correct within 1 g.

(*d*) *Measure.*—A calibrated cylin-

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates.

[2] Accepted by the Society at annual meeting, June, 1942.

drical measure of suitable size, preferably of 0.1 cu. ft. capacity, fitted with a flat glass cover $\frac{3}{8}$ in. or more in thickness and having a diameter or width 1 to 2 in. greater than the diameter of the measure.

(*e*) *Thermometer.*—A calibrated thermometer, such that readings in the neighborhood of room temperature will be correct within 0.2 C.

(*f*) *Syringe.*—A rubber syringe for adjusting water levels and for rinsing.

(*g*) *Spray Nozzle.*—A fine spray nozzle (insecticide type) on a hose attached to a water tap.

Calibration of Pycnometer and Measure

3. (*a*) The pycnometer and measure, cleaned and dried, shall each be weighed to the nearest gram. The pycnometer shall be filled with freshly boiled distilled water to the level of the gage point in the manner described in Section 5 (*b*), and the weight of the water and its temperature determined. The weight of the water divided by its density (as determined from physical tables) gives the volume of the pycnometer. This volume, to the nearest 1 ml., shall be taken as the average of at least three independent determinations.

(*b*) The volume of the measure shall be determined in a manner similar to that described in Paragraph (*a*), but the measure shall be filled slightly overfull, the excess water being removed by placing the glass cover plate on the measure and moving the plate back and forth. After weighing, the temperature of the water shall be determined, and the volume of the measure shall be calculated as the weight of water divided by its density.

Determination of Density of Laboratory Water

4. The water supply in many laboratories may be appreciably heavier than distilled water, and the comparatively large quantity involved in this method may require an appreciable correction for the density as taken from the physical tables. For this determination the calibrated pycnometer shall be used. The weight of the tap water required to fill the pycnometer to the gage point level shall be taken as the average of at least three independent determinations, the temperature being closely observed each time. The weight of the water divided by the volume of the pycnometer gives the density, and this shall be compared with the density of distilled water at the same temperature. The difference constitutes a density correction that shall be applied in all calculations involving the density of the laboratory water.

Procedure

5. (*a*) A sample of the concrete to be tested shall be placed in the calibrated measure in the standard manner (Note). The measure shall be just perceptibly overfilled, and the excess removed by sliding the glass cover across the top with a back and forth motion. Care shall be taken to see that the concrete is everywhere in contact with the glass and that the measure is exactly full. The "bulk" weight of the concrete shall then be obtained to the nearest 1 g.

NOTE.—For comparative purposes, the standard method of filling the measure shall be that described in Section 5 of the Standard Method of Test for Weight per Cubic Foot, Yield, and Air Content (Gravimetric) of Concrete (A.S.T.M. Designation: C 138) of the American Society for Testing Materials,[3] except that if the air content in concrete placed by vibration or in any other specific manner is to be determined, the sample shall be placed in the measure in that manner.

(*b*) The concrete in the measure shall be returned to the batch, remixed therewith, and a representative portion scooped into the pycnometer until it

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

is slightly more than half full. The pycnometer shall then be weighed and immediately filled with water at room temperature, to a level an inch or two below that of the gaging point. The cover shall be clamped on, and the concrete thoroughly mixed with the water by first rotating the pycnometer from end to end and then rolling it slowly back and forth on the edge of the table. The mixing operation, designed to remove all the entrained air, shall be continued for a period of 3 to 5 min. The pycnometer shall then be slowly upended, the cover removed, and any adhering solids rinsed back into the pycnometer. If any appreciable amount of foam or scum remains on the surface of the water, it shall be thoroughly sprayed and reduced to minimum volume by a fine water spray. Small residual amounts of foam or scum shall then be removed from the surface by means of the syringe, care being taken to remove none of the cement in the operation. The pycnometer shall then be placed on a smooth and level table, and the water brought to the level of the gage point. The proper level shall be determined by raising and lowering the glass plate without jarring, while adjusting the water level, until the meniscus just persists in forming when the plate rests on the rim of the pycnometer. If a trace of foam or scum remaining on the surface of the water interferes with precise gaging, a clear surface may be obtained by touching it with the tip of the finger moistened with kerosine. When the gaging is completed, the gage shall be removed, and the pycnometer shall be cleaned and dried on the exterior and weighed to the nearest 1 g.

(*c*) To insure that the air is completely removed, the pycnometer shall be resealed with the cover, and the operation of shaking, rolling, and redetermining the water level shall be repeated. If after this operation the weight of the system does not increase by more than 2 g., the test shall be concluded by determining the temperature of the water in the pycnometer as closely as the thermometer can be read. If the gain in weight is more than 2 g., the operation of shaking, rolling, and regaging shall again be repeated.

Calculation

6. The air content of the concrete shall be calculated as follows:

$$B = \frac{W_p}{W_m} \times V_m$$

$$C = V_p - V_w$$

$$A = \frac{B - C}{B} \times 100$$

where:

A = percentage of air contained in the concrete,
B = bulk volume in milliliters of the sample weighed in the pycnometer,
C = volume in milliliters of the sample weighed in the pycnometer minus the volume of the entrained air,
W_p = weight in grams of the sample weighed in the pycnometer,
W_m = weight in grams of the sample in the filled calibrated measure,
V_m = volume in milliliters of the calibrated measure,
V_p = volume in milliliters of the pycnometer, and
V_w = volume in milliliters of the water required to fill the pycnometer after the de-airing process.

NOTE.—The following example is submitted as a guide to the sequence of observations and computations, showing data and calculations from a determination of the air content of a laboratory batch of hand-mixed concrete. All weights are in grams and all volumes are in milliliters.

(1) Weight of cement	1150
(2) Weight of fine aggregate (saturated and surface dry)	2275
(3) Weight of coarse aggregate (saturated and surface dry)	4225
(4) Weight of water	680
(5) Weight of batch, (1) + (2) + (3) + (4)	8330
(6) Weight of 0.1-cu. ft. measure	4232.5
(7) Weight of 0.1-cu. ft. measure and concrete	10913
(8) Temperature of concrete, deg. C.	19.8
(9) Weight of concrete, (7) − (6)	6680.5
(10) Weight of pycnometer	4440.5
(11) Weight of pycnometer and concrete	10015
(12) Weight of pycnometer, concrete, and water to fill	11780
(13) Temperature of water, deg. C.	19.3
(14) Repeat (12)	11779.5
(15) Repeat (12) again (if necessary)	
(16) Temperature of water, deg. C.	19.5
(17) Density of water, (from tables, corrected)	0.9987
(18) Weight of concrete, (11) − (10)	5574.5
(19) Weight of water, (14) − (11) or (15) − (11)	1764.5
(20) Volume of water, (19) ÷ (17)	1766.8
(21) Volume of pycnometer (from calibration)	4104
(22) Absolute volume of concrete, (21) − (20)	2337.2
(23) Volume of 0.1-cu. ft. measure (from calibration)	2830
(24) Bulk volume of concrete, (23) × (18) ÷ (9)	2361.5
(25) Volume of air, (24) − (22)	24.3
(26) Percentage of air, 100 × (25) ÷ (24)	1.03
(27) Absolute specific gravity of concrete, (18) ÷ (22)	2.3
(28) Bulk specific gravity of concrete, (9) ÷ (23)	2.3

Tentative Method of

MAKING AND CURING CONCRETE COMPRESSION AND FLEXURE TEST SPECIMENS IN THE LABORATORY[1]

A.S.T.M. Designation: C 192 – 44 T

ISSUED, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. This method covers the procedure for making and curing compression and flexure test specimens of concrete in the laboratory under accurate control of quantities of materials and test conditions.

NOTE.—For the method of making and curing compression and flexure test specimens of concrete during construction, see the Standard Method of Making and Curing Concrete Compression and Flexure Test Specimens in the Field (A.S.T.M. Designation: C 31).[3]

Preparation of Materials

2. (*a*) Materials shall be brought to room temperature (preferably 65 to 75 F., 18 to 24 C.) before beginning the tests.

(*b*) *Cement.*—Cement shall be stored in a dry place, in moisture-proof containers; preferably made of metal. The cement shall be thoroughly mixed, in order that the sample may be uniform throughout the tests. It shall be passed through a No. 16 (1190-micron) sieve and all lumps rejected.

(*c*) *Aggregates.*—Aggregates for each batch of concrete shall be of the desired gradation. In general, coarse aggregates shall be separated into two or more size fractions, depending upon the maximum size of aggregate used, and recombined for each batch in such a manner as to produce the desired gradation. Fine aggregates shall be separated into different sizes if unusual gradations are being studied. Aggregates shall be treated before use to insure a definite and uniform condition of moisture by one of the three following procedures:

(*1*) They shall be brought to a saturated, surface-dry condition.

(*2*) They shall be brought to a saturated condition with surface moisture in sufficiently small amounts to preclude loss by draining and shall be so maintained until used. When using this method the amounts of surface moisture on the coarse and fine aggregates shall be determined prior to making concrete specimens.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates.

[2] This method represents a revision of and replaces the procedures for making and curing specimens which appeared in the former Standard Method of Test for Compressive Strength of Concrete (C 39 – 42) and the Standard Method of Test for Flexural Strength of Concrete (Laboratory Method Using Simple Beam with Third Point Loading) (C 78 – 39).

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(3) The aggregates in a saturated condition shall be immersed in water and shall be weighed under water. The required immersed weight may be calculated as follows:

$$W_w = \frac{W_a(G - 1)}{G}$$

where:

W_a = desired weight in air of the aggregate in a saturated condition,
G = bulk specific gravity of the aggregate in a saturated condition, and
W_w = weight in water.

Upon removal of the aggregate from the water, an additional weighing in air of aggregate and surface water will be necessary to determine the amount of surface water in the aggregate.

Weighing Materials

3. All materials shall be weighed on scales meeting the requirements for sensibility reciprocal[4] and tolerances prescribed by the National Bureau of Standards.[5] Noncompensating spring scales shall not be used. Where the scales are graduated in decimals of a pound instead of ounces, or where the metric system is used, the equivalent percentage sensibility reciprocal and tolerances shall apply.

NOTE.—The user is cautioned against making small weighings on large capacity scales.

Mixing Concrete

4. (a) *General.*—Concrete shall be mixed either by hand or in a suitable laboratory mixer in batches of such size as to leave about 10 per cent excess after molding test specimens.

(b) *Hand Mixing.*—The batch shall be mixed in a watertight, clean, damp, metal pan, with either a blunted bricklayer's trowel or a shovel, whichever is more convenient, using the following procedure:

(1) The cement and fine aggregate shall be mixed until they are thoroughly blended.

(2) The coarse aggregate shall be added and the entire batch mixed until the coarse aggregate is uniformly distributed throughout the batch.

(3) Water shall be added and the mass mixed until the concrete is homogeneous in appearance and has the desired consistency. If prolonged mixing is required, because of the addition of water in increments while adjusting the slump, the batch shall be discarded and a new batch made without interrupting the mixing to make trial slump tests.

(c) *Machine Mixing.*—The procedure specified for hand mixing shall be followed unless a different procedure is better adapted to the mixer being used. If necessary to eliminate segregation, machine-mixed concrete shall be deposited in a watertight, clean, damp, metal pan and remixed by shovel or trowel.

Consistency and Yield of Concrete

5. (a) *Consistency.*—The consistency of each batch of concrete shall be measured immediately after mixing, by one or both of the following methods of the American Society for Testing Materials:

(1) *Slump Test.* Standard Method of Slump Test for Consistency of Portland-Cement Concrete (A.S.T.M. Designation: C 143).[3]

(2) *Flow Test.* Standard Method of Test for Flow of Portland-Cement

[4] The sensibility reciprocal is a measure of the sensitiveness of a balance, and is the weight required to move the position of the pointer one division. For a complete definition of sensibility reciprocal, see "Specifications, Tolerances, and Regulations for Commercial Weighing and Measuring Devices," *Handbook H29*, Nat. Bureau Standards, September, 1942, pp. 87 and 88.

[5] "Specifications, Tolerances and Regulations for Commercial Weighing and Measuring Devices," *Handbook H29*, Nat. Bureau Standards, Section I-2, p. 122 and Section J-2, p. 130.

Concrete by Use of the Flow Table (A.S.T.M. Designation: C 124).[3]

(*b*) *Yield.*—The yield of each batch of concrete shall be determined by the Standard Method of Test for Weight per Cubic Foot, Yield, and Air Content (Gravimetric) of Concrete (A.S.T.M. Designation: C 138).[3]

(*c*) All concrete used for consistency or yield tests shall be returned to the mixing pan and the entire batch remixed just enough to produce a homogeneous mass.

Number of Specimens

6. Three or more test specimens for each variable shall be made for each period or condition of test. Specimens involving any given variable in the mix shall be made from at least three separate batches. An equal number of specimens for each variable shall be made on any given day. When it is impossible to make at least one specimen for each variable in the mix on a given day, the mixing of the entire series of specimens shall be completed in as few days as possible and one of the mixes shall be repeated each day as a standard of comparison.

NOTE.—Test periods of 7 and 28 days are recommended for compression tests. Flexure specimens are frequently tested at 14 and 28 days. For longer test periods, 3 months and 1 yr. are recommended.

COMPRESSION TEST SPECIMENS

Size of Specimens

7. Compression test specimens shall be cylindrical with a length equal to twice the diameter. Standard cylindrical specimens shall be 6 in. in diameter by 12 in. in length if the coarse aggregate does not exceed 2 in. in nominal size. Smaller test specimens shall have a ratio of diameter of specimen to maximum size aggregate of not less than 3 to 1, except that the diameter of the specimen shall not be less than 3 in. for mixtures containing aggregate more than 5 per cent of which is retained on a No. 4 (4760-micron) sieve. For concrete containing aggregates larger than 2 in. nominal size, cylindrical specimens shall have a diameter at least three times the maximum nominal size of aggregate. The oversize of any nominal size aggregate used shall not exceed the requirements prescribed in the Standard Specifications for Concrete Aggregates (A.S.T.M. Designation: C 33).[3]

Molds

8. Molds for compression test specimens shall be of metal and shall be provided with a machined metal base plate. Means shall be provided for securing the base plate to the mold. The assembled mold and base plate shall be watertight and shall be oiled with mineral oil before use.

NOTE.—Satisfactory molds may be made from cold drawn, seamless steel tubing or from steel pipe machined on the inside. These tubular sections shall be cut to the proper length, split along one element and fitted with a circumferential metal band and bolt for closing. Satisfactory molds may also be made from iron or steel castings. In general, molds made from formed sheet metal are not satisfactory.

Molding Specimens

9. The test specimens shall be formed by placing the concrete in the mold in three layers of approximately equal volume. In placing each scoopful of concrete, the scoop shall be moved around the top edge of the mold as the concrete slides from it in order to insure a symmetrical distribution of the concrete within the mold. The concrete shall be further distributed by a circular motion of the tamping rod. Each layer shall be rodded with 25 strokes of a tamping rod, $\frac{5}{8}$-in. in diameter and approximately 24 in. in length, bullet-

shaped at the lower end. The strokes shall be distributed uniformly over the cross-section of the mold and shall penetrate into the underlying layer. The bottom layer shall be rodded throughout its depth. After the top layer has been rodded, the surface of the concrete shall be struck off with a trowel and covered with a glass or metal plate to prevent evaporation.

Capping Specimens

10. (*a*) The test specimens may be capped with a thin layer of stiff, neat-cement paste after the concrete has ceased settling in the molds, generally from 2 to 4 hr. or more after molding. The cap shall be formed by means of plate glass not less than $\frac{1}{4}$ in. in thickness or a machined metal plate not less than $\frac{1}{2}$ in. in thickness and having a minimum surface dimension at least 1 in. larger than the diameter of the mold. It shall be worked on the cement paste until its lower surface rests on top of the mold. The cement for capping shall be mixed to a stiff paste 2 to 4 hr. before it is to be used in order to avoid the tendency of the cap to shrink. Adhesion of the concrete to the top and bottom plates may be avoided by coating them with oil or grease.

(*b*) Specimens not capped with neat-cement paste as described in Paragraph (*a*) shall be ground or capped before testing. In all cases the capped or ground surface shall not depart from a plane by more than 0.001 in. and shall be at right angles to the axis of the specimen. Caps shall be made as thin as practicable and shall not flow or fracture when the specimen is tested.

NOTE.—Neat portland or alumina cement and suitable mixtures of sulfur with granular materials are recognized as suitable for capping hardened concrete specimens. Sulfur caps should be allowed to harden for at least 1 hr. before applying load; cement caps for a sufficient period to comply with the requirements prescribed in Paragraph (*b*).

Curing Specimens

11. The test specimens shall be removed from the molds not earlier than 20 hr. nor later than 48 hr. after molding and stored in a moist condition (Note 1) at a temperature within the range of 65 to 75 F. (18 to 24 C.) (Note 2) until the time of test. Specimens shall not be exposed to a stream of running water. If storage in water is desired, a saturated lime solution shall be used.

NOTE 1.—Moist condition is that in which free water is maintained on the surfaces of the specimens at all times.

NOTE 2.—Attention is directed to the fact that the temperature within damp sand and under wet burlap or similar materials will always be lower than the temperature in the surrounding atmosphere if evaporation takes place.

FLEXURE TEST SPECIMENS

Size of Specimens

12. The cross-section of the flexure test specimen shall be 6 by 6 in. if the coarse aggregate does not exceed 2 in. in nominal size. For larger coarse aggregate, the minimum cross-sectional dimension shall be not less than three times the maximum nominal size of the coarse aggregate. The oversize of any nominal size used shall not exceed the requirements prescribed in the Standard Specifications for Concrete Aggregate (A.S.T.M. Designation: C 33).[3]

Molds

13. Molds for flexure test specimens shall be rigid and nonabsorptive and shall be at least 3 in. longer than the required span length as prescribed in Section 3 of the Standard Method of Test for Flexural Strength of Concrete (Using Simple Beam with Third-Point Loading) (A.S.T.M. Designation: C 78).[3] Means shall be provided for securing the base plate to the mold. The assembled mold and base plate shall be watertight and shall be lightly oiled with a mineral oil before use.

Molding Specimens

14. The test specimen shall be formed with its long axis horizontal. The concrete shall be placed in layers approximately 3 in. in depth and each layer shall be rodded 50 times for each square foot of area. The top layer shall slightly overfill the mold. After each layer is rodded, the concrete shall be spaded along the sides and ends with a mason's trowel or other suitable tool. When the rodding and spading operations are completed, the top shall be struck off with a straightedge and finished with a wood float. The test specimen shall be made promptly and without interruption and covered with a double layer of wet burlap, which shall be kept wet until the specimen is removed from the mold. While in the molds, the specimens shall be kept within the temperature range specified in Section 2. (*a*).

Curing Specimens

15. Flexure test specimens shall be cured as prescribed in Section 11.

Tentative Specifications for

WATERPROOF PAPER FOR CURING CONCRETE[1]

A.S.T.M. Designation: C 171 - 42 T

ISSUED, 1942.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These specifications cover waterproof paper intended for use as a curing material for portland cement concrete.

General Requirements

2. Waterproof paper shall consist of two sheets of plain kraft paper cemented together with a bituminous material in which are embedded cords or strands of fiber running in both directions of the paper, not more than $1\frac{1}{4}$ in. apart. The paper shall be light in color, shall be free of visible defects, and shall have a uniform appearance. It shall be sufficiently strong and tough to permit its use under the conditions existing on highway and structural work without tearing or otherwise becoming unfit for the use for which it is intended.

Impermeability

3. Mortar specimens, when covered with the paper to be tested and tested in accordance with the Tentative Method of Test for Water Retention Efficiency of Methods for Curing Concrete (A.S.T.M. Designation: C 156) of the American Society for Testing Materials,[3] shall not show a moisture loss greater than 10 per cent of the original mixing water used. In the test the paper shall be placed on the mortar specimen immediately after molding and shall remain in place for three days.

Tensile Strength

4. The paper shall conform to the following requirements as to tensile strength:

		Machine Direction	Cross Direction
Tensile strength, lb. per in. of width	dry	50	30
	wet	20	10

Tensile Strength Test

Apparatus

5. The testing machine shall consist of the following:

(*a*) *Clamps.*—Two clamps whose centers shall be in the same plane parallel with the direction of motion of the stressing clamp and so aligned that they will hold the test specimen in one plane.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates.

[2] Accepted by the Society at annual meeting, June, 1942.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(*b*) *Pendulum.*—A pendulum so attached to one clamp as to accurately balance the load applied to the test specimen.

(*c*) *Scale.*—A device attached to the pendulum to indicate on a graduated scale the breaking load of the test specimen. The scale shall be graduated in weight units which may be read to an accuracy of not less than 0.2 per cent of the total reading.

(*d*) *Loading Device.*—A means of moving the stressing clamp at a uniform rate. The capacity of the machine shall be such that the tensile strength of the paper to be tested will be not greater than 90 per cent nor less than 10 per cent of the capacity of the machine. The machine shall preferably be power driven.

Calibration of Apparatus

6. The testing machine shall be accurately leveled in both of the principal directions. The stressing clamp shall be displaced or removed and accurate weights corresponding to various divisions of the scale markings shall be suspended from the pendulum actuating clamp. The weights shall be held at the start and released slowly so that the pendulum is actuated at a rate similar to that specified in Section 9 (*b*), and other conditions shall be as nearly as possible the same as when a specimen is being tested. A record shall be made of deviations from the scale readings and corresponding corrections shall be made in the test results. The machine shall be calibrated at intervals of not more than 30 days.

Test Specimens

7. Specimens for test shall be cut accurately, and not less than ten from each principal direction of the paper shall be so selected as to be representative of the sample. The specimens shall be cut exactly 2 in. in width and the edges shall be clean-cut and parallel.

Conditioning

8. (*a*) Specimens to be tested dry shall be preconditioned in air maintained at a temperature between 70 and 75 F. and at a relative humidity of 50 per cent, for a period of 12 hr. prior to testing.

(*b*) Specimens to be tested wet shall be immersed in water having a temperature between 70 and 75 F., for a period of 1 hr. immediately prior to testing.

Procedure

9. (*a*) All tensile strength tests shall be made in an atmosphere maintained at a temperature of 70 to 75 F. and at a relative humidity of 50 per cent.

(*b*) The distance between the jaws of the testing machine at the beginning of the test shall be 7 in. The test specimen shall be firmly clamped squarely in the jaws of the clamps and the stressing jaw then operated at a speed of 12 ± 2 in. per min. until the specimen breaks. The breaking load shall be recorded to the nearest 2 per cent of the total indicated reading. Not less than ten specimens cut in each principal direction of the paper shall be tested. All readings obtained when the paper breaks at or in the jaws shall be rejected.

Report

10. The tensile strength results in pounds per inch of width shall be reported to the nearest 2 per cent of the total reading. The average, maximum, and minimum tensile strengths for each of the principal directions of the paper shall be reported.

Tentative Method of Test for

WATER RETENTION EFFICIENCY OF METHODS FOR CURING CONCRETE[1]

A.S.T.M. Designation: C 156 - 44 T

ISSUED, 1940; REVISED, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. This method is intended for laboratory use in determining the efficiency of methods used for curing concrete, as measured by their ability to prevent moisture loss during the early hardening period.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Molds.*—Molds shall be made of metal, glass, hard rubber, or a plastic material and shall be watertight. They shall be rigidly constructed to prevent distortion and shall be thoroughly cleaned before each use. The molds shall be 6 by 12 in. at the top, $5\frac{3}{4}$ by $11\frac{3}{4}$ in. at the bottom, and $2\frac{1}{8}$ in. in depth, measured on the inside.

(*b*) *Curing Cabinet.*—A cabinet for curing the specimens at a temperature of 100 ± 2 F. and a relative humidity of 32 ± 2 per cent.

Test Specimens

3. Test specimens shall be approximately 6 in. in width by 12 in. in length by 2 in. in depth.

Control Specimens

4. For each series of test runs, a series of specimens made and tested in accordance with this method shall be run concurrently with the regular test specimens (Section 3), except that no curing agent shall be applied to the exposed surface.

Proportioning and Mixing Mortar

5. (*a*) *Proportioning.*—Mortar for the specimens shall be of plastic consistency. The proportions of cement (Note 1) and sand shall be determined by adding dry sand (Note 2) to a cement paste having a water-cement ratio of 0.40 by weight, to produce a flow of 50 ± 5. The flow test shall be made on the 10-in. flow table, using ten $\frac{1}{2}$-in. drops in 10 sec. (Note 3).

NOTE 1.—Portland cement shall conform to the Standard Specifications for Portland Cement (A.S.T.M. Designation: C 150).[3] Type I portland cement without an air-entraining agent shall be used.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates.

[2] Revision accepted by the Society at annual meeting, June, 1944.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

NOTE 2.—The sand for making test specimens shall be natural silica sand from Ottawa, Ill., graded as follows:

Sieve Size	Percentage Retained
No. 100 (149-micron)	98 ± 2
No. 50 (297-micron)	72 ± 5
No. 30 (590-micron)	2 ± 2
No. 16 (1190-micron)	None

NOTE 3.—The flow test shall be made as described in the Standard Method of Test for Structural Strength of Fine Aggregate Using Constant Water-Cement-Ratio Mortar (A.S.T.M. Designation: C 87)[3] except that the mold shall be filled in two equal layers and each layer puddled 25 times with the gloved finger.

(*b*) *Mixing.*—The mortar shall be mixed preferably at room temperature of 70 ± 5 F, and at a relative humidity of 40 to 60 per cent. The temperature of the mortar at the time of molding shall be 70 ± 2 F. Cement and water in quantities which will give a water-cement ratio of 0.40 by weight shall be placed in a nonabsorbent vessel and the cement permitted to absorb water for 1 min. These materials shall then be mixed with a spoon into a smooth paste. Sand from a sample of known weight shall be added to the paste until the mortar has the desired consistency. Final mixing shall be accomplished by continuous kneading and squeezing with the hands for 2 min. Rubber gloves shall be worn during the mixing operation.

Molding Specimens

6. A layer of mortar shall be placed in a weighed mold to a depth of approximately 1 in. and puddled with the gloved fingers until uniformly distributed. A second 1-in. layer of mortar shall then be placed in the mold and puddled in a similar manner. Immediately after puddling the second layer, the surface of the specimen shall be screeded with a wood template cut to strike off $\frac{3}{8}$ in. below the top of the mold. The screed or template shall have a flat screeding surface 1 in. in width by approximately 5 in. in length. One pass only shall be made in the direction of the long axis of the specimen, using a sawing motion of the screed.

Number of Specimens

7. A set of three or more test specimens shall be made in order to constitute a test of a given curing method. A control specimen shall be made for each regular test specimen except where two or more curing methods are being investigated concurrently in the same cabinet, in which case only one set of three or more control specimens need be made.

Storage of Specimens

8. (*a*) Immediately after molding, the mold and the specimen shall be weighed to the nearest gram and placed in the curing cabinet in which the atmosphere shall be maintained at a temperature of 100 ± 2 F. and a relative humidity of 32 ± 2 per cent. The specimen shall be level and not subjected to vibration. Spacing between the individual specimens and between the specimens and the side walls of the cabinet shall be so arranged as to provide a clear space on all sides of from 2 to 7 in. Within these limits the spacing shall be the same for all specimens in the cabinet. In order to provide for uniform air distribution, dummy molds shall be used to fill the shelf of the cabinet when it is not filled with test specimens.

(*b*) Air shall be circulated below the specimens in a horizontal plane at a velocity of 6 ± 2 miles per hr. The main movement of conditioned air within the cabinet shall be in a vertical plane. This shall be accomplished by exhausting air from the top of the cabinet and admitting conditioned air below the specimens. Sufficient space shall be provided in the cabinet above the test specimens so that the moving air will not be deflected in such manner as to pass over the speci-

mens in a horizontal plane. Air shall not be blown directly across the top of the specimens. In cases where the curing materials under test contain volatile solvents, means shall be provided for introducing fresh air in such quantities that a complete change of air within the cabinet is accomplished at least twice during the first 12 hr. and at least once every 24 hr. thereafter for the duration of the test. All specimens shall be stored on the same shelf, or on a series of shelves, provided the conditions of air circulation for each shelf are obtained independently of any other shelf.

Application of Curing Materials

9. (*a*) The specimen shall be removed from the cabinet when the surface water has disappeared and it is possible to lightly brush the surface without smearing or drawing more water to the surface. Immediately upon removal from the curing cabinet, the specimen shall be weighed and the edges sealed by means of a suitable sealing compound that will not be affected by the curing material under test.[4] The sealing compound shall effectively seal against moisture loss between the boundary of the specimen and mold, and shall not extend more than $\frac{1}{4}$ in. onto the surface of the specimen.

(*b*) After sealing, the mortar surface shall be lightly brushed in parallel strokes with a stiff bristle brush to break up laitance and remove air bubbles. Another weight determination shall then be made.

(*c*) The curing material shall then be uniformly applied to the surface of the specimen at the rate of coverage specified for that method of curing. It is advisable to apply curing agents to only one specimen at a time in an atmosphere of controlled temperature and humidity as prescribed in Section 5 (*b*). Liquids shall be sprayed on. The proper coverage shall be determined by weighing the spray gun before and after application to 0.1 g.

(*d*) Paper, blankets or burlap shall receive an additional sealing where they come in contact with the mold in order to prevent moisture loss except through the curing medium. In cases where a blanket having a thickness greater than $\frac{1}{8}$ in. is used, the exposed edges, where they project above the mold, shall also be sealed. Liquid curing materials or granular curing materials shall be applied uniformly.

(*e*) Blankets or burlap intended for field use in a continuously wet condition shall be kept wet during the entire period of test. Similar materials intended for field use with but one initial wetting shall be applied in a wet condition but shall not be subsequently wetted during the period of test.

(*f*) Where the method under investigation involves the use of a combination of curing agents, such as the use of a membrane or paper following a preliminary wet curing, the time of application of the final curing agent shall be in accordance with the governing specifications.

(*g*) After each treatment or stage of treatment the specimens shall be weighed. Additional weighings shall be made 3 hr. after application and daily for the duration of the test. The weights at 3 hr. will give an indication of any unusual loss caused by a leaking mold or faulty seal. The specimens shall be returned to the curing cabinet without delay.

Duration of Test

10. The test shall extend over a period of 3 days.

[4] A mixture consisting of four parts Bexin resin and one part Vinsol resin by weight has been found satisfactory for this purpose. The mixture shall be heated to a fluid consistency and thoroughly mixed prior to use.

Corrections for Loss in Weight of Liquid Curing Materials During Test

11. The loss in weight of volatile matter from a liquid curing material shall be determined by coating a metal pan or plate (with edges raised $\frac{1}{8}$ in.) of an area equal to the top of test specimen with the same quantity of curing material as used on the specimen. The pan or plate shall be placed in the curing cabinet with the liquid-cured specimen and weighed each time the specimen is weighed. If the pans or plates reach constant weight, no further weighings are necessary. The loss in weight of the liquid curing material shall be applied as a correction before calculating the moisture loss.

Calculation of Loss in Weight

12. At the end of the 3-day curing period the mold and specimen shall be weighed and the loss in weight, based on the weight of the specimen immediately after molding, shall be calculated. This loss shall be corrected by applying a correction for the volatile matter lost from the curing material as determined in Section 11, and by the weight of the sealing compound and curing material added.

Calculation of Percentage Loss of Moisture

13. From the proportionate parts of materials going into the mix, the weight of water in the specimen at the time of molding shall be calculated. The percentage loss of moisture shall be determined by dividing the loss in weight as determined in Section 12 by the weight of water in the specimen at the time of molding as determined above and multiplying by 100. The percentage loss of moisture shall be calculated for both the test specimens and the control specimens. Specimens varying in moisture loss by more than 3 per cent from the average of all specimens constituting a test, shall be discarded when calculating the average moisture loss.

Calculation of Efficiency Index

14. The efficiency index shall be calculated as follows:

$$E = \frac{W - W_1}{W} \times 100$$

where:

E = efficiency index,
W = average percentage loss of moisture by control specimens, and
W_1 = average percentage loss of moisture by test specimens.

Tentative Specifications for

STANDARD SIZES OF COARSE AGGREGATE FOR HIGHWAY CONSTRUCTION[1]

A.S.T.M. Designation: D 448 – 42 T

ISSUED, 1937; REVISED, 1941, 1942.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These specifications cover standard size designations and maximum permissible ranges in mechanical analyses for standard sizes of coarse aggregate and screenings for use in the construction or maintenance of various types of highways and highway structures.

Manufacture

2. The standard sizes of coarse aggregate prescribed in these specifications may be manufactured by means of any suitable commercial process and by the use of any sizes or shapes of plant screen openings necessary to produce the designated sizes within the limits of the mechanical analyses specified in Section 3.

Standard Sizes

3. Standard sizes of coarse aggregate shall comply with the sizes given in Table I. All sizes shall be determined by means of laboratory sieves having square openings and conforming to the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials.[3]

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] Revision accepted by Committee E-10 on Standards, August 24, 1942.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

TABLE I.—STANDARD SIZES OF COARSE AGGREGATE.

Size Number	Nominal Size, Square Openings	Amounts Finer than Each Laboratory Sieve (Square Openings), per cent by weight													
		4 in.	3½ in.	3 in.	2½ in.	2 in.	1½ in.	1 in.	¾ in.	½ in.	⅜ in.	No. 4 (4760-micron)	No. 8 (2380-micron)	No. 16 (1190-micron)	No. 100 (149-micron)
1	3½ to 1½ in.	100	90 to 100	....	25 to 60	....	0 to 15	....	0 to 5	....	....	....	....	....	....
2	2½ to 1½ in.	....	....	100	90 to 100	35 to 70	0 to 15	....	0 to 5	....	....	....	....	....	....
24	2½ to ¾ in.	....	....	100	90 to 100	....	25 to 60	....	0 to 10	0 to 5	....	....	....	....	....
3	2 to 1 in.	....	....	....	100	90 to 100	35 to 70	0 to 15	....	0 to 5	....	....	....	....	....
357	2 in. to No. 4	....	....	....	100	95 to 100	....	35 to 70	....	10 to 30	....	0 to 5	....	....	....
4	1½ to ¾ in.	....	....	....	....	100	90 to 100	20 to 55	0 to 15	....	0 to 5	....	....	....	....
467	1½ in. to No. 4	....	....	....	....	100	95 to 100	....	35 to 70	....	10 to 30	0 to 5	....	....	....
5	1 to ⅜ in.	....	....	....	....	....	100	90 to 100	40 to 75	15 to 35	0 to 15	0 to 5	....	....	....
57	1 in. to No. 4	....	....	....	....	....	100	90 to 100	....	25 to 60	....	0 to 10	0 to 5	....	....
6	¾ to ⅜ in.	....	....	....	....	....	....	100	90 to 100	20 to 55	0 to 15	0 to 5	....	....	....
67	¾ in. to No. 4	....	....	....	....	....	....	100	90 to 100	....	20 to 55	0 to 10	0 to 5	....	....
68	¾ in. to No. 8	....	....	....	....	....	....	100	90 to 100	....	30 to 65	5 to 25	0 to 5	....	....
7	½ in. to No. 4	....	....	....	....	....	....	....	100	90 to 100	40 to 70	0 to 15	0 to 5	....	....
78	½ in. to No. 8	....	....	....	....	....	....	....	100	90 to 100	40 to 75	5 to 25	0 to 5	....	....
8	⅜ in. to No. 8	....	....	....	....	....	....	....	....	100	85 to 100	10 to 30	0 to 10	....	....
89	⅜ in. to No. 16	....	....	....	....	....	....	....	....	100	90 to 100	20 to 55	5 to 30	0 to 1	....
9	No. 4 to No. 16	....	....	....	....	....	....	....	....	....	100	85 to 100	10 to 40	0 to 10	....
10	No. 4 to 0[a]	....	....	....	....	....	....	....	....	....	100	85 to 100	....	....	10 to 30

[a] Screenings.

Tentative Specifications for

MATERIALS FOR STABILIZED BASE COURSE[1]

A.S.T.M. Designation: D 556 – 40 T

ISSUED, 1940.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. (*a*) These specifications cover the quality and size of sand-clay mixtures, gravel, stone or slag screenings, sand, crusher-run coarse aggregate consisting of gravel, crushed stone, or slag combined with soil mortar, or any combination of these materials for use in the construction of a stabilized base course. The requirements are intended to cover only materials having normal or average specific gravity, absorption, and gradation characteristics. When materials such as caliche, gypsum, limerock, and water-soluble salts are to be used, appropriate limits suitable to their use must be specified.

(*b*) The following types of base course stabilized mixtures are covered:

Type A.—Sand-clay mortar.

Type B.—Graded coarse aggregate.

Type C.—Gravel, stone or slag screenings, or sand.

General Requirements

2. (*a*) *Type A.*—Type A material shall consist of natural or artificial mixtures of clay or soil binder with gravel, sand, or other aggregate so proportioned as to conform to the requirements prescribed in these specifications. The aggregate retained on the No. 10 (2000-micron) sieve shall consist of hard, durable particles and shall be free from injurious or deleterious substances.

(*b*) *Type B.*—Type B material shall consist of natural or artificial mixtures of gravel, stone, or slag and soil mortar so proportioned as to conform to the requirements prescribed in these specifications. The coarse aggregate shall consist of clean, hard, durable particles of crushed or uncrushed gravel, stone, or slag free from soft, thin, elongated, or laminated pieces, and from roots and other vegetable matter, or other deleterious substances. It shall be hard and durable enough to resist weathering, traffic abrasion, and crushing. Shale and similar materials that break up and weather rapidly when alternately frozen and thawed or wetted and dried should not be used. The soil mortar shall be that portion passing the No. 10 (2000-micron) sieve and shall consist of soil binder and granular material such as stone or slag screenings or sand.

(*c*) *Type C.*—Type C material shall

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A. S. T. M. Committee D-4 on Road and Paving Materials.

[2] Accepted by the Society at annual meeting, June, 1940. Editorially revised in 1942.

consist of gravel, stone or slag screenings, or sand, or mixtures thereof so proportioned as to conform to the requirements prescribed in these specifications. The material shall consist of hard, durable particles, free from injurious or deleterious substances, uniformly graded from coarse to fine.

Sieve Analysis

3. The material shall conform to the following requirements:

TYPE A

Sieve	Percentage Passing
1-in.	100
No. 10 (2000-micron)	65 to 100

The material passing the No. 10 sieve shall conform to the following requirements:

Sieve	Percentage Passing
No. 10 (2000-micron)	100
No. 20 (840-micron)	55 to 90
No. 40 (420-micron)	35 to 70
No. 200 (74-micron)	8 to 25[a]

TYPE B

Sieve	Percentage Passing	
	B-1 1-in. max. size	B-2 2-in. max. size
2-in.		100
1½-in.		70 to 100
1-in.	100	55 to 85
¾-in.	70 to 100	50 to 80
⅜-in.	50 to 80	40 to 70
No. 4 (4760-micron)	35 to 65	30 to 60
No. 10 (2000-micron)	25 to 50	20 to 50
No. 40 (420-micron)	15 to 30	10 to 30
No. 200 (74-micron)	5 to 15[a]	5 to 15[a]

TYPE C

Sieve	Percentage Passing
¾-in.	100
No. 4 (4760-micron)	70 to 100
No. 10 (2000-micron)	35 to 80
No. 40 (420-micron)	25 to 50
No. 200 (74-micron)	8 to 25[a]

[a] The percentage passing the No. 200 sieve shall be not more than one-half of that passing the No. 40 sieve.

Liquid Limit

4. The material passing the No. 40 (420-micron) sieve shall have a liquid limit not exceeding 25.

Plasticity Index

5. The material passing the No. 40 (420-micron) sieve shall have a plasticity index of not more than 6 for types A and B, and of not more than 3 for type C.

Moisture Content

6. Materials of types A, B, and C shall contain sufficient moisture to insure maximum compaction.

Admixtures

7. Chemicals or other admixtures shall conform to the specifications of the American Society for Testing Materials that are applicable. When the chemical to be used is not covered by A. S. T. M. specifications, a good commercial grade meeting the approval of the engineer shall be used.

Methods of Testing

8. The material shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(*a*) *Sampling.*—Tentative Methods of Sampling Stone, Slag, Gravel, Sand, and Stone Block for Use as Highway Materials (A. S. T. M. Designation: D 75).[3]

(*b*) *Sieve Analysis.*—Standard Method of Test for Sieve Analysis of Fine and Coarse Aggregates (A. S. T. M. Designation: C 136).[3]

(*c*) *Liquid Limit.*—Standard Method of Test for Liquid Limit of Soils (A. S. T. M. Designation: D 423).[3]

(*d*) *Plasticity Index.*—Standard Method of Test for Plastic Limit and Plasticity Index of Soils (A. S. T. M. Designation: D 424).[3]

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Tentative Specifications for
MATERIALS FOR STABILIZED SURFACE COURSE[1]

A.S.T.M. Designation: D 557 – 40 T

ISSUED, 1940.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. (*a*) These specifications cover the quality and size of sand-clay mixtures, gravel, stone or slag screenings, sand, crusher-run coarse aggregate consisting of gravel, crushed stone, or slag combined with soil mortar, or any combination of these materials for use in the construction of a stabilized surface course. The requirements are intended to cover only materials having normal or average specific gravity, absorption, and gradation characteristics. When materials such as caliche, gypsum, limerock, and water-soluble salts are to be used, appropriate limits suitable to their use must be specified.

(*b*) The following types of surface course stabilized mixtures are covered:

Type A.—Sand-clay mortar.

Type B.—Graded coarse aggregate.

Type C.—Gravel, stone or slag screenings, or sand.

General Requirements

2. (*a*) *Type A.*—Type A material shall consist of natural or artificial mixtures of clay or soil binder with gravel, sand, or other aggregate so proportioned as to conform to the requirements prescribed in these specifications. The aggregate retained on the No. 10 (2000-micron) sieve shall consist of hard, durable particles and shall be free from injurious or deleterious substances.

(*b*) *Type B.*—Type B material shall consist of natural or artificial mixtures of gravel, stone, or slag with soil mortar, so proportioned as to conform to the requirements prescribed in these specifications. The coarse aggregate shall consist of clean, hard, durable particles of crushed or uncrushed gravel, stone, or slag free from soft, thin, elongated, or laminated pieces, and from roots and other vegetable matter, or other deleterious substances. It shall be hard and durable enough to resist weathering, traffic abrasion, and crushing. Shale and similar materials that break up and weather rapidly when alternately frozen and thawed or wetted and dried shall not be used. The soil mortar shall be that portion passing the No. 10 (2000-micron) sieve and shall consist of soil binder and granular material such as stone or slag screenings or sand.

(*c*) *Type C.*—Type C material shall

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A. S. T. M. Committee D-4 on Road and Paving Materials.

[2] Accepted by the Society at annual meeting, June, 1940. Editorially revised in 1942.

consist of gravel, stone or slag screenings, or sand, or mixtures thereof so proportioned as to conform to the requirements prescribed in these specifications. The material shall consist of hard, durable particles, free from injurious or deleterious substances, uniformly graded from coarse to fine.

Sieve Analysis

3. The material shall conform to the following requirements:

TYPE A

Sieve	Percentage Passing
1-in.	100
No. 10 (2000-micron)	65 to 100

The material passing the No. 10 sieve shall conform to the following requirements:

Sieve	Percentage Passing
No. 10 (2000-micron)	100
No. 20 (840-micron)	55 to 90
No. 40 (420-micron)	35 to 70
No. 200 (74-micron)	8 to 25[a]

TYPE B

Sieve	Percentage Passing
1-in.	100
½-in.	85 to 100
⅜-in.	65 to 100
No. 4 (4760-micron)	55 to 85
No. 10 (2000-micron)	40 to 70
No. 40 (420-micron)	25 to 45
No. 200 (74-micron)	10 to 25[a]

TYPE C

Sieve	Percentage Passing
½-in.	100
No. 4 (4760-micron)	70 to 100
No. 10 (2000-micron)	35 to 80
No. 40 (420-micron)	25 to 50
No. 200 (74-micron)	8 to 25[a]

[a] The percentage passing the No. 200 sieve shall be not more than two-thirds of that passing the No. 40 sieve.

Liquid Limit

4. The material passing the No. 40 (420-micron) sieve shall have a liquid limit not exceeding 35.

Plasticity Index

5. The material passing the No. 40 (420-micron) sieve shall have a plasticity index of not less than 4 nor more than 9.

Moisture Content

6. Materials of types A, B, and C shall contain sufficient moisture to insure maximum compaction.

Admixtures

7. Chemicals or other admixtures shall conform to the specifications of the American Society for Testing Materials that are applicable. When the chemical to be used is not covered by A. S. T. M. specifications, a good commercial grade meeting the approval of the engineer shall be used.

Methods of Testing

8. The material shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(*a*) *Sampling.*—Tentative Methods of Sampling Stone, Slag, Gravel, Sand, and Stone Block for Use as Highway Materials (A. S. T. M. Designation: D 75).[3]

(*b*) *Sieve Analysis.*—Standard Method of Test for Sieve Analysis of Fine and Coarse Aggregates (A. S. T. M. Designation: C 136).[3]

(*c*) *Liquid Limit.*—Standard Method of Test for Liquid Limit of Soils (A. S. T. M. Designation: D 423).[3]

(*d*) *Plasticity Index.*—Standard Method of Test for Plastic Limit and Plasticity Index of Soils (A. S. T. M. Designation: D 424).[3]

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Tentative Specifications for

CRUSHED STONE, CRUSHED SLAG, AND GRAVEL FOR BITUMINOUS CONCRETE BASE AND SURFACE COURSES OF PAVEMENTS[1]

A.S.T.M. Designation: D 692 - 42 T

ISSUED, 1942.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These specifications cover the quality and grading of crushed stone, crushed slag, and gravel suitable for use as coarse aggregate in the following types of bituminous concrete mixtures for base and surface courses of pavements:

Open-mix bituminous concrete base course,

Closed-mix bituminous concrete base course,

Bituminous concrete binder course,

Coarse-graded bituminous concrete surface course,

Fine-graded bituminous concrete surface course (modified Topeka, or stone-filled sheet asphalt).

Basis of Purchase

2. In adapting these specifications to any particular locality or project, the type or types of aggregate (crushed stone, crushed slag, or gravel) to be supplied shall be specified.

General Characteristics

3. (*a*) *Crushed Stone.*—The crushed stone shall consist of clean, tough, durable fragments free of an excess of thin or elongated pieces, and reasonably free of soft or disintegrated pieces, stone coated with dirt, or other objectionable matter.

(*b*) *Crushed Slag.*—The crushed slag shall be air-cooled blast-furnace slag and shall consist of fragments reasonably uniform in density and quality and reasonably free of thin or elongated pieces, dirt, or other objectionable matter.

(*c*) *Gravel.*—The gravel shall consist of clean, tough, durable pieces free of an excess of thin or elongated pieces, and reasonably free of soft or disintegrated pieces, gravel coated with dirt, or other objectionable matter. The gravel shall be crushed and uncrushed, as specified in Section 5(*b*). Crushed gravel shall consist of fragments having two or more fractured surfaces.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] Accepted by Committee E-10 on Standards, August 24, 1942.

These specifications are in effect a revision and consolidation of, and replace the former Standard Specifications for Gravel for Bituminous Base (D 309 - 30) and the Tentative Specifications for Crushed Slag for Bituminous Concrete Base and Surface Courses (D 485 - 38 T) and for Crushed Stone for Bituminous Concrete Base and Surface Courses (D 486 - 39 T), which specifications were accordingly discontinued in 1942.

Physical Properties

4. (*a*) *Wear.*—Coarse aggregate shall conform to the following requirements for resistance to wear:

	Coarse Aggregate for Use in Base Courses	Coarse Aggregate for Use in Binder and Surface Courses
Wear, Los Angeles abrasion machine, max., per cent	50	40

(*b*) *Unit Weight of Slag.*—Crushed slag shall conform to the following requirements for unit weight, compacted:

	Crushed Slag for Use in Base Courses	Crushed Slag for Use in Binder and Surface Courses
Weight per cubic foot, compacted, min., lb	65	70

NOTE.—The requirements for physical properties of coarse aggregate incorporated in these specifications are recommended as the minimum requirements for quality. It is not expected, however, that these limits for quality will be applicable to all localities and conditions.

Sizes

5. (*a*) The sizes of coarse aggregate for the various uses shall conform to the following requirements (Notes 1 to 3):

	Size Number
Open-mix bituminous concrete base course	24, 3, 4, 5[a]
Closed-mix bituminous concrete base course	357, 467, 57
Bituminous concrete binder course	57, 67
Coarse-graded bituminous concrete surface course	5, 57, 67
Fine-graded bituminous concrete surface course	78, 8
Seal coat	78, 8, 9

[a] Size No. 5 aggregate for open-mix bituminous concrete base course shall be either crushed stone or crushed slag.

NOTE 1.—An appropriate size should be selected for each use, depending on local construction and service conditions. Sizes have been selected from the Tentative Specifications for Standard Sizes of Coarse Aggregate for Highway Construction (A.S.T.M. Designation: D 448) of the American Society for Testing Materials.[3]

NOTE 2.—The size of aggregate for seal coat, when a seal coat is specified, should be adapted to the maximum size of coarse aggregate in the bituminous concrete surface mixture. The following combinations of sizes are recommended:

Coarse Aggregate Size	Seal-Coat Aggregate Size
5 or 57	78
67	8
78 or 8	9

NOTE 3.—When asphalt mixing plants are suitably equipped for control of aggregate gradings and proportions, other appropriate sizes may be selected from Table I of the Tentative Specifications for Standard Sizes of Coarse Aggregate for Highway Construction (A.S.T.M. Designation: D 448) of the American Society for Testing Materials.[3] The selected sizes should be such that when combined in suitable proportions, they will produce the required grading in the finished mixtures.

(*b*) *Crushed Pieces in Gravel.*—Gravel shall conform to the following requirements as to percentage of crushed pieces:

	Crushed Pieces, min., per cent
Open-mix bituminous concrete base course	75
Closed-mix bituminous concrete base course	50
Bituminous concrete binder course	50
Coarse-graded bituminous concrete surface course	50
Fine-graded bituminous concrete surface course	0
Seal coat	0

Sieve Analysis

6. The several sizes of coarse aggregate, when tested by means of laboratory sieves having square openings, shall conform to the requirements shown in Table I.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Methods of Sampling and Testing

7. The coarse aggregate shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(*a*) *Sampling.*—Tentative Methods of Sampling Stone, Slag, Gravel, Sand, and Stone Block for Use as Highway Materials (A.S.T.M. Designation: D 75).[3]

(*b*) *Wear.*—Standard Method of Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine (A.S.T.M. Designation: C 131).[3]

(*c*) *Weight per Cubic Foot.*—Standard Method of Test for Unit Weight of Aggregate (A.S.T.M. Designation: C 29).[3]

TABLE I.–GRADING REQUIREMENTS.

Size Number	Nominal Size (Sieves with Square Openings)	Amounts Finer than Each Laboratory Sieve (Square Openings), per cent by weight										
		3 in.	2½ in.	2 in.	1½ in.	1 in.	¾ in.	½ in.	⅜ in.	No. 4 (4760-micron)	No. 8 (2380-micron)	No. 16 (1190-micron)
24	2½ to ¾ in.	100	90 to 100	...	25 to 60	...	0 to 10	0 to 5	...	...	...	...
3	2 to 1 in.	...	100	90 to 100	35 to 70	0 to 15	...	0 to 5	...	...	...	...
357	2 in. to No. 4	...	100	95 to 100	...	35 to 70	...	10 to 30	...	0 to 5	...	...
4	1½ to ¾ in.	...	...	100	90 to 100	20 to 55	0 to 15	...	0 to 5	...	...	...
467	1½ in. to No. 4	...	...	100	95 to 100	...	35 to 70	...	10 to 30	0 to 5	...	...
5	1 to ⅜ in.	...	...	...	100	90 to 100	40 to 75	15 to 35	0 to 15	0 to 5	...	...
57	1 in. to No. 4	...	...	...	100	90 to 100	...	25 to 60	...	0 to 10	0 to 5	...
67	¾ in. to No. 4	...	...	...	...	100	90 to 100	...	20 to 55	0 to 10	0 to 5	...
78	¾ in. to No. 8	...	...	...	...	...	100	90 to 100	40 to 75	5 to 25	0 to 5	...
8	⅜ in. to No. 8	...	...	...	...	...	...	100	85 to 100	10 to 30	0 to 10	...
9	No. 4 to No. 16	...	...	...	...	...	...	...	100	85 to 100	10 to 40	0 to 10

(*d*) *Sieve Analysis.*—Standard Method of Test for Sieve Analysis of Fine and Coarse Aggregates (A.S.T.M. Designation: C 136).[3]

Tentative Methods of

SAMPLING STONE, SLAG, GRAVEL, SAND, AND STONE BLOCK FOR USE AS HIGHWAY MATERIALS[1]

A.S.T.M. Designation: D 75 – 42 T

ISSUED, 1939; REVISED, 1940, 1942.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These methods are intended to apply to the sampling of stone, slag, gravel, sand, and stone block for the following purposes:

Preliminary investigation of sources of supply,

Acceptance or rejection of source of supply,

Inspection of shipments of materials, and

Inspection of materials on the site of the work.

Securing Samples

2. (*a*) Samples of all materials for test upon which is to be based the acceptance or rejection of the supply shall be taken by the purchaser or his authorized representative. Samples for inspection or preliminary test may be submitted by the seller or owner of the supply.

(*b*) Sampling is equally as important as the testing, and the sampler shall use every precaution to obtain samples that will show the true nature and condition of the materials which they represent.

STONE FROM LEDGES OR QUARRIES

Inspection

3. The ledge or quarry face of the stone shall be inspected to determine any variation in different strata. Differences in color and structure shall be observed.

Sampling and Size of Sample

4. Separate samples of stone weighing at least 50 lb. each of unweathered specimens shall be obtained from all strata that appear to vary in color and structure. When the toughness or compression test is required, one piece of each sample shall be not smaller than 4 by 5 by 3 in. in size with the bedding plane plainly marked, and this piece shall be free of seams or fractures. Pieces that have been damaged by blasting shall not be included in the sample.

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] Revision accepted by Committee E-10 on Standards, August 24, 1942.

Prior to their present publication as tentative, these methods were published as standard from 1922 to 1939, but withdrawn and republished as tentative in 1939.

Record

5. In addition to the general information accompanying all samples, the following information shall accompany samples from local ledges that are not commercial sources:

(*1*) Name of owner or seller,

(*2*) Approximate quantity available (if quantity is very large this can be recorded as practically unlimited),

(*3*) Quantity and character of overburden or stripping,

(*4*) Haul to nearest point on road where the material is to be used,

(*5*) Character of haul (kind of road and grade), and

(*6*) Some detailed record of the extent and location of the material represented by each sample.

NOTE.—A sketch, plan and elevation, showing the thickness and location of the different layers is recommended for this purpose.

FIELD STONE AND BOULDERS

Inspection

6. A detailed inspection of the deposits of field stone and boulders, over the area where the supply is to be obtained, shall be made. The different kinds of stone and their condition in the various deposits shall be recorded.

Sampling

7. Separate samples shall be selected of all classes of stone that visual inspection indicates would be considered for use in construction.

Record

8. Records accompanying samples of field stone and boulders, in addition to general information, shall contain the following:

(*1*) Location of supply,

NOTE.—The plotting of the field stone and boulder area on a U. S. topographic or a similar map is recommended for this purpose.

(*2*) Approximate quantity available, and

(*3*) The percentages of different classes of stone that were sampled, and the percentages of material that can be rejected by visual examination and may therefore have to be handled and rejected.

SAND AND GRAVEL

Roadside Productions

Description of Term

9. Roadside production shall be understood to be the production of materials with portable or semiportable crushing, screening, or washing plants established or reopened in the vicinity of the work on a designated project for the purpose of supplying materials for that project.

Sampling

10. (*a*) Samples shall be so chosen as to represent the different materials that are available in the deposit. An estimate of the quantity of the different materials shall be made.

(*b*) If the deposit is worked as an open face bank or pit, the sample shall be taken by channeling the face so that it will represent material that visual inspection indicates may be used. Care shall be observed to eliminate any material that has fallen from the face along the surface. It is necessary, especially in small deposits, to excavate test holes some distance back of and parallel to the face to determined the extent of the supply. The number and depth of these test holes depend on the quantity of material that is to be used from the deposit. Material that would be stripped from the pit as overburden, etc., shall not be included in the sample. Separate samples shall be obtained from the face of the bank and from test holes; and, if visual inspection indicates that there is considerable variation in the material, separate samples shall be

obtained at different depths. If information on the variations in the pit is desired, each of the samples shall be tested, but if the average condition only is desired, the separate samples may be mixed into a composite sample and reduced by quartering to the size required for test. If the material being sampled consists of sand, a sample weighing at least 25 lb. shall be obtained. If the material being sampled consists of a mixture of sand and gravel, the sample shall be large enough to yield not less than 25 lb. of the lesser constituent.

(*c*) Deposits that have no open face shall be sampled by means of test holes. The number and depth of these test holes will depend on local conditions and the amount of material to be used from the deposit. A separate sample shall be obtained from each test hole and if information on the variations in the deposit is desired, each of these samples shall be tested, but if the average condition only is desired and visual examination indicates no radical difference in size of grain, color, etc., the separate samples may be mixed into a composite sample and reduced by quartering to the size required for test.

(*d*) It is very difficult to secure a representative sample from a stock pile and if conditions require sampling from this source, it is recommended that separate samples be taken from different parts of the pile, care being taken to observe any segregated areas and bearing in mind that the material near the base of the pile is likely to be segregated and coarser than the average of the material in the pile. In sampling sand, the outer layer of material shall be removed until damp sand is reached.

Record

11. In addition to the general information accompanying all samples from roadside productions, the detailed information prescribed in items 1 to 6 of Section 5 shall be supplied.

SAND, GRAVEL, STONE, AND SLAG

Commercial Sources

Sampling for Quality

12. (*a*) Where practicable, samples from commercial sources shall be obtained from the finished product. Otherwise the sample shall be taken in accordence with the procedure described in Section 10.

(*b*) Samples to be tested for abrasion loss by the Standard Method of Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine (A.S.T.M. Designation: C 131) of the American Society for Testing Materials[3] shall be obtained from commercially prepared material.

Sampling at Plant

13. A general inspection of the plant and a record of the screening facilities shall be made. The sample shall preferably be obtained from cars or boats during the loading from stock piles or bins. In order to determine variations in the grading of the material, separate samples shall be obtained at different times while the material is being loaded. If the samples are obtained from a bin, they shall be taken from the entire cross-section of the flow of material as it is being discharged. Approximately 2 to 5 tons of material should be allowed to flow from the bin before the sample is obtained. The testing of separate samples will give a better idea of the variations that occur but samples shall be mixed and reduced by quartering when the average condition is desired

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Sampling at Delivery

14. (*a*) Where it is not practicable to visit the plant, samples for both quality and size may be obtained at the destination, preferably while the material is being unloaded. The sampler should realize that segregation of different sizes is very likely to occur, and samples shall be so chosen as to show any differences which occur, both in quality and size of material. Separate samples shall be taken from the top, middle, and bottom of each unit of the shipment. These separate samples shall be mixed to form a composite sample and this sample shall, if necessary, be reduced by quartering, but if information on variation is desired, the separate samples shall be tested.

(*b*) Where test is to be made for size only, it is recommended that tests be made in the field in order not to delay decision on the use of the material. Samples shall also be sent to the laboratory for check tests.

Number and Size of Samples

15. (*a*) The number of samples required depends on the intended use of the material, the quantity of material involved, and the variations both in quality and size of the aggregate. A sufficient number of samples shall be obtained to cover all variations in the material. It is recommended that each sample of crushed stone, gravel, slag, or sand represent approximately 50 tons of material.

(*b*) Samples of crushed stone, gravel, slag, and sand which are to be subjected to a mechanical analysis in accordance with the Standard Method of Test for Sieve Analysis of Fine and Coarse Aggregates (A.S.T.M. Designation: C 136) of the American Society for Testing Materials[a] shall conform to the weight requirements prescribed in Table I.

TABLE I.—SIZE OF SAMPLES.

Nominal Maximum Size of Particles, Passing Sieve	Minimum Weight of Field Samples, lb.	Minimum Weight of Sample for Test,[a] g.
FINE AGGREGATE		
No. 10	10	100
No. 4	10	500
COARSE AGGREGATE		
⅜ in.	10	1 000
½ in.	20	2 500
¾ in.	30	5 000
1 in.	50	10 000
1½ in.	70	15 000
2 in.	90	20 000
2½ in.	100	25 000
3 in.	125	30 000
3½ in.	150	35 000

[a] The sample for test shall be obtained from the field sample by quartering or other suitable means to insure a representative portion.

BANK RUN SAND AND GRAVEL

Size of Samples

16. (*a*) Samples of run of bank (where the sand and gravel are combined) shall weigh at least 100 lb. when the gravel content is 50 per cent or more of the whole. If the gravel is less in percentage, the sample shall be increased in proportion.

NOTE: *Example.*—When the gravel percentage is 25 per cent of the whole, the sample should weigh 200 lb.

(*b*) Samples for mechanical analysis shall conform to the requirements for size of sample as prescribed in Table I.

MISCELLANEOUS MATERIALS

Sampling

17. Samples of slag sand, stone sand, screenings, mine tailings, and all other materials used instead of sand and gravel or broken stone, shall be inspected in the same manner and samples shall be taken in the same manner as prescribed for the materials of similar size and classification.

STONE BLOCK

Place of Sampling

18. Samples of stone block shall be taken either at the quarry or at the destination as directed by the purchaser. Blocks that would be rejected by visual inspection shall not be included in the sample.

Size of Sample

19. The sample shall consist of at least six blocks and the bedding plane shall be marked on at least two of these.

MARKING AND SHIPPING SAMPLES

Marking

20. Each sample or separate container shall be accompanied by a card or regular form, preferably in the container, giving the following information:

(*1*) By whom taken, and the official title or rank of the sampler,

(*2*) By whom submitted,

(*3*) Source of supply, and in case of commercial supplies, daily production,

(*4*) Proposed use for the material, and

(*5*) Geographic location, and shipping facilities (name of railroad, canal or river, or other common carrier).

Shipping Samples

21. (*a*) *Stone and Slag.*—Samples of ledge stone, crushed stone, and slag shall be shipped in a secure box or bag.

(*b*) *Gravel, Sand, etc.*—Samples of run of bank gravel, sand, screenings, and other fine material, shall be shipped in a tight box or closely woven bag so there will be no loss of the finer particles.

(*c*) *Stone Block.*—Samples of stone block shall be securely crated.

Tentative Method of Test for

ABRASION OF GRAVEL BY USE OF THE DEVAL MACHINE[1]

A.S.T.M. Designation: D 289 - 42 T

ISSUED, 1928; REVISED, 1937; WITHDRAWN, 1940; REISSUED AS TENTATIVE WITHOUT REVISION, 1942.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. This method covers the procedure for testing uncrushed or crushed gravel for resistance to abrasion in the Deval testing machine with an abrasive charge. Uncrushed gravel consists entirely of uncrushed fragments and, for the purpose of this test, a gravel containing more than 10 per cent by weight of crushed fragments shall be considered as crushed gravel.

Apparatus

2. (*a*) *Deval Machine.*—The Deval abrasion testing machine shall consist of one or more hollow cast iron cylinders closed at one end and furnished with a tightly-fitting iron cover at the other. The inside dimensions of the cylinders shall be 20 cm. in diameter and 34 cm. in depth. These cylinders shall be mounted on a shaft at an angle of 30 deg. with the axis of rotation of the shaft.

(*b*) *Sieve.*—The No. 12 (1680-micron) square-hole sieve shall conform to the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials.[3]

Abrasive Charge

3. (*a*) The abrasive charge shall consist of 6 cast iron spheres or steel spheres approximately $1\frac{7}{8}$ in. in diameter, each weighing between 390 and 445 g. The cast iron spheres shall conform to the following requirements as to chemical composition:

Combined carbon, min., per cent	2.50
Graphitic carbon, max., per cent	0.25
Manganese, max., per cent	0.50
Phosphorus, max., per cent	0.25
Sulfur, max., per cent	0.08
Silicon, max., per cent	1.00

NOTE.—Cast iron spheres, uniform in size, conforming to these specifications are available. The above chemical requirements for the cast iron spheres are identical with those for the cast iron spheres used in the rattler test for paving brick as prescribed in the Standard Specifications for Paving Brick (A.S.T.M. Designation: C 7) of the American Society for Testing Materials.[3]

[1] Under the standardization procedure of the Society, this method is under the joint jurisdiction of the A.S.T.M. Committees C-9 on Concrete and Concrete Aggregates and Committee D-4 on Road and Paving Materials.

[2] Accepted by the Society at annual meeting, June, 1942.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Steel spheres $1\frac{27}{32}$ in. in diameter and weighing 417 g. can be obtained from at least one manufacturer. If they are not readily available, the next larger size ($1\frac{7}{8}$ in.) may be ground to size.

(*b*) An abrasive charge of 6 spheres weighing 2500 ± 10 g. shall be used with each test sample.

Grading

4. The gravel shall be separated by screening into the various sizes required for grading the test sample according to one of the gradings specified in Section 5. The material of these sizes shall be washed and dried.

Test Sample

5. (*a*) The test sample shall consist of dry gravel made up of percentages of the various sizes conforming to one of the gradings shown in the following table. The grading used shall be that most nearly representing the gravel furnished for the work.

Grading	Passing Sieve	Retained on Sieve	Percentage of Sample
A	$\frac{3}{4}$-in.	$\frac{1}{2}$-in.	25
	1-in.	$\frac{3}{4}$-in.	25
	$1\frac{1}{2}$-in.	1-in.	25
	2-in.	$1\frac{1}{2}$-in.	25
B	$\frac{3}{4}$-in.	$\frac{1}{2}$-in.	25
	1-in.	$\frac{3}{4}$-in.	25
	$1\frac{1}{2}$-in.	1-in.	50
C	$\frac{3}{4}$-in.	$\frac{1}{2}$-in.	50
	1-in.	$\frac{3}{4}$-in.	50
D	$\frac{3}{4}$-in.	$\frac{1}{2}$-in.	50
	$\frac{3}{4}$-in.	$\frac{1}{2}$-in.	50

(*b*) The weight of the test sample shall depend upon its average bulk specific gravity and shall be as follows:

Range in Bulk Specific Gravity	Weight of Sample, g.
Over 2.8	5500
2.4 to 2.8	5000
2.2 to 2.39	4500
Less than 2.2	4000

(*c*) When the gravel furnished for the work contains as much as 25 per cent of material finer than $\frac{1}{2}$ in. but is of such size that either grading *A*, *B*, or *C* would be used for the abrasion test, a second abrasion test shall be made using grading *D*, if, in the opinion of the engineer, the particles less than $\frac{1}{2}$ in. in size are not at least equal in hardness to those particles $\frac{1}{2}$ in. or over in size.

(*d*) *Crushed Gravel.*—Gravel containing more than 10 per cent by weight of crushed fragments (Note) shall, for the purpose of this test be considered as crushed gravel. In such cases, the test sample shall contain crushed fragments so as to be representative of the gravel furnished for the work and shall be prepared in accordance with Sections 4 and 5.

NOTE.—For the purpose of this test, a crushed gravel fragment may be considered as a fragment of gravel having at least one fractured face.

Procedure

6. The test sample and the abrasive charge shall be placed in the Deval abrasion testing machine and the machine rotated for 10,000 revolutions at a speed of from 30 to 33 rpm. At the completion of the test, the material shall be removed from the machine and sieved on a No. 12 (1680-micron) sieve. The material retained on the sieve shall be washed, dried, and accurately weighed to the nearest gram.

Calculations

7. (*a*) *Uncrushed Gravel.*—In the case of uncrushed gravel, the loss by abrasion shall be considered as the difference between the original weight of the test sample and the weight of the material retained on the No. 12 (1680-micron) sieve, expressed as a percentage of the original weight of the test sample.

(*b*) *Crushed Gravel.*—In the case of crushed gravel, the percentage by weight of crushed fragments shall be determined,

and the permissible percentage of wear which shall govern shall be calculated as follows:

$$W = \frac{AL + (100 - A)\,L'}{100}$$

where:

W = permissible percentage of wear,
A = percentage of uncrushed fragments,
$100 - A$ = percentage of crushed fragments,
L = maximum percentage of wear permitted by the specifications for gravel consisting entirely of uncrushed fragments, and
L' = maximum percentage of wear permitted by the specifications for gravel consisting entirely of crushed fragments.

Tentative Specifications for

CUT-BACK ASPHALT[1]

RAPID CURING TYPE

A.S.T.M. Designation: D 597 - 40 T

ISSUED, 1940.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These specifications cover liquid petroleum products, produced by fluxing an asphaltic base with a suitable light volatile solvent, to be used in the treatment of road surfaces in the manner designated for the respective grades.

Properties

2. The cut-back asphalt shall be free from water and shall conform to the requirements prescribed in Table I.

Methods of Testing

3. The material shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials, except as specified in Paragraph (*a*):

(*a*) *Flash Point (Tag Open Cup).*—Standard Method of Test for Flash Point with Tagliabue Open Cup (Method T 79) of the American Association of State Highway Officials.[3]

(*b*) *Viscosity, Saybolt Furol.*—Standard Method of Test for Viscosity by Means of the Saybolt Viscosimeter (A.S.T.M. Designation: D 88).[4]

(*c*) *Distillation.*—Standard Method of Test for Distillation of Cut-Back Asphaltic Products (A.S.T.M. Designation: D 402).[4]

NOTE.—If a 100-ml. graduate does not permit sufficiently close readings to determine conformity to specifications with the desired accuracy, receivers graduated with 0.1-ml. divisions shall be used.

(*d*) *Penetration.*—Standard Method of Test for Penetration of Bituminous Materials (A.S.T.M. Designation: D 5).[4]

(*e*) *Ductility.*—Standard Method of Test for Ductility of Bituminous Materials (A.S.T.M. Designation: D 113).[4]

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] Accepted by Committee E-10 on Standards, August 28, 1940.

[3] See "Standard Specifications for Highway Materials and Methods of Sampling and Testing," The Am. Assn. State Highway Officials, Part II, p. 92 (1942).

[4] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(*f*) *Solubility in Carbon Tetrachloride.*—Method No. 1 of the Standard Method of Test for Determination of Bitumen (A.S.T.M. Designation: D 4),[4] except that c.p. carbon tetrachloride shall be used in place of carbon disulfide.

TABLE I.—REQUIREMENTS FOR CUT-BACK ASPHALT (RAPID CURING TYPE).

Designation	Grade RC-0		Grade RC-1		Grade RC-2		Grade RC-3		Grade RC-4		Grade RC-5	
Uses	Note 1		Notes 1, 2, 4		Notes 1, 2, 3, 4		Notes 1, 2, 4		Notes 1, 4		Notes 1, 4, 5	
	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.
Flash point (Tag open cup), deg. Fahr.	...	...	...	...	80	...	80	...	80	...	80	...
Viscosity, Saybolt Furol:												
at 77 F. (25 C.), sec.	75	150	...	...	...	...	...	...	...	...	...	...
at 122 F. (50 C.), sec.	...	...	75	150	...	...	...	...	...	...	...	...
at 140 F. (60 C.), sec.	...	...	...	...	100	200	250	500	...	...	...	...
at 180 F. (82.2 C.), sec.	...	...	...	...	...	...	...	...	125	250	300	600
Distillation test:												
Distillate, percentage by volume of total distillate to 680 F. (360 C.):												
to 374 F. (190 C.)	15	...	10	...	...	...	...	...	...	...	...	...
to 437 F. (225 C.)	55	...	50	...	40	...	25	...	8	...	...	...
to 500 F. (260 C.)	75	...	70	...	65	...	55	...	40	...	25	...
to 600 F. (316 C.)	90	...	88	...	87	...	83	...	80	...	70	...
Residue from distillation to 680 F. (360 C.), percentage volume by difference	50	...	60	...	67	...	73	...	78	...	82	...
Tests on residue from distillation:												
Penetration at 77 F. (25 C.), 100 g., 5 sec.	80	120	80	120	80	120	80	120	80	120	80	120
Ductility at 77 F. (25 C.), cm.	100	...	100	...	100	...	100	...	100	...	100	...
Solubility in carbon tetrachloride, per cent	99.5	...	99.5	...	99.5	...	99.5	...	99.5	...	99.5	...

NOTE 1.—Used as binder in surface treatments.
NOTE 2.—Used as binder in road-mix construction with open-graded aggregate.
NOTE 3.—Used as binder in cold patch mixtures with open-graded aggregate.
NOTE 4.—Used as binder in cold-laid plant-mix construction with open-graded aggregate.
NOTE 5.—Used as binder in penetration macadam construction under cold weather conditions.

Tentative Specifications for

CUT-BACK ASPHALT[1]

MEDIUM CURING TYPE

A.S.T.M. Designation: D 598 - 40 T

ISSUED, 1940.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These specifications cover liquid petroleum products, produced by fluxing an asphaltic base with suitable distillates, to be used in the treatment of road surfaces in the manner designated for the respective grades.

Properties

2. The cut-back asphalt shall be free from water and shall conform to the requirements prescribed in Table I.

Methods of Testing

3. The material shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials, except as specified in Paragraph (*a*):

(*a*) *Flash Point (Tag Open Cup).*—Standard Method of Test for Flash Point with Tagliabue Open Cup (Method T 79) of the American Association of State Highway Officials.[3]

(*b*) *Viscosity, Saybolt Furol.*—Standard Method of Test for Viscosity by Means of the Saybolt Viscosimeter (A.S.T.M. Designation: D 88).[4]

(*c*) *Distillation.*—Standard Method of Test for Distillation of Cut-Back Asphaltic Products (A.S.T.M. Designation: D 402).[4]

NOTE.—If a 100-ml. graduate does not permit sufficiently close readings to determine conformity to specifications with the desired accuracy, receivers graduated with 0.1-ml. divisions shall be used.

(*d*) *Penetration.*—Standard Method of Test for Penetration of Bituminous Materials (A.S.T.M. Designation: D 5).[4]

(*e*) *Ductility.*—Standard Method of Test for Ductility of Bituminous Materials (A.S.T.M. Designation: D 113).[4]

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] Accepted by Committee E-10 on Standards, August 28, 1940.

[3] See "Standard Specifications for Highway Materials and Methods of Sampling and Testing," The Am. Assn. State Highway Officials, Part II, p. 92 (1942).

[4] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(*f*) *Solubility in Carbon Tetrachloride.*—Method No. 1 of the Standard Method of Test for Determination of Bitumen (A.S.T.M. Designation: D 4),[4] except that c.p. carbon tetrachloride shall be used in place of carbon disulfide.

TABLE I.—REQUIREMENTS FOR CUT-BACK ASPHALT (MEDIUM CURING TYPE).

Designation	Grade MC-0		Grade MC-1		Grade MC-2		Grade MC-3		Grade MC-4		Grade MC-5	
Uses	Note 1		Note 1		Notes 1, 2, 4, 6		Notes 2, 3, 4, 5, 7		Notes 2, 3, 5, 7		Notes 2, 7	
	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.
Flash point (Tag open cup), deg. Fahr.	100	...	100	...	150	...	150	...	150	...	150	...
Viscosity, Saybolt Furol:												
at 77 F. (25 C.), sec.	75	150	...	...	...	...	...	...	...	...	...	...
at 122 F. (50 C.), sec.	...	...	75	150	...	...	...	...	...	...	...	...
at 140 F. (60 C.), sec.	...	...	...	...	100	200	250	500	...	...	...	...
at 180 F. (82.2 C.), sec.	...	...	...	...	...	...	...	...	125	250	300	600
Distillation test:												
Distillate, percentage by volume of total distillate to 680 F. (360 C.):												
to 437 F. (225 C.)	...	25	...	20	...	10	...	5	...	0	...	0
to 500 F. (260 C.)	40	70	25	65	15	55	5	40	...	30	...	20
to 600 F. (316 C.)	75	93	70	90	60	87	55	85	40	80	20	75
Residue from distillation to 680 F. (360 C.), percentage volume by difference	50	...	60	...	67	...	73	...	78	...	82	...
Tests on residue from distillation:												
Penetration at 77 F. (25 C.), 100 g., 5 sec.	120	300	120	300	120	300	120	300	120	300	120	300
Ductility at 77 F. (25 C.), cm.[a]	100	...	100	...	100	...	100	...	100	...	100	...
Solubility in carbon tetrachloride, per cent	99.5	...	99.5	...	99.5	...	99.5	...	99.5	...	99.5	...

[a] If penetration of residue is more than 200 and its ductility at 77 F. is less than 100, the material will be acceptable if its ductility at 60 F. is more than 100.

NOTE 1.—Used as priming material.
NOTE 2.—Used as binder in surface treatments.
NOTE 3.—Used as binder in road-mix construction with open-graded aggregate.
NOTE 4.—Used as binder in road-mix construction with dense-graded aggregate.
NOTE 5.—Used as binder in cold patch mixtures with open-graded aggregate.
NOTE 6.—Used as binder in cold patch mixtures with dense-graded aggregate.
NOTE 7.—Used as binder in cold-laid plant-mix construction with dense-graded aggregate.

Tentative Specifications for

TAR[1]

A.S.T.M. Designation: D 490 - 43 T

ISSUED, 1938; REVISED, 1943.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These specifications cover 14 grades of tar, as follows:

Grades RT-1, RT-2, RT-3, and RT-4 for use in prime coat application and light surface treatment,

Grades RT-5 and RT-6 for use in surface treatment and road mix,

Grades RT-7, RT-8, and RT-9 for use in surface treatment, road mix, plant mix, and seal coat,

Grades RT-10, RT-11, and RT-12 for use in surface treatment, plant mix, penetration, crack filler, and seal coat, and

Grades RT.C.B.-5 and RT.C.B.-6 for use in patching, surface treatment; also for plant mix and road mix where low-temperature application and quick setting are desired.

Properties

2. The tar shall conform to the requirements prescribed in Table I.

Methods of Sampling and Testing

3. The material shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials, with the exception of the tests specified in Paragraphs (*d*) and (*h*):

(*a*) *Sampling.*—Tentative Methods of Sampling Bituminous Materials (A.S.T.M. Designation: D 140).[3]

(*b*) *Water.*—Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (A.S.T.M. Designation: D 95).[3]

(*c*) *Specific Gravity.*—Standard Method of Test for Specific Gravity of Road Oils, Road Tars, Asphalt Cements, and Soft Tar Pitches (A.S.T.M. Designation: D 70).[3]

(*d*) *Specific Viscosity.*—Standard Method of Test for Specific Viscosity (Engler) (Method T 54)[4] of the American Association of State Highway Officials. The results shall be reported as specific viscosity compared with water at 25 C. (77 F.).

(*e*) *Float Test.*—Standard Method of

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] Revision accepted by Committee E-10 on Standards, November 10, 1943.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

[4] See "Standard Specifications for Highway Materials and Methods of Sampling and Testing," The Am. Assn. State Highway Officials, Part II, p. 55 (1942).

TABLE 1.—REQUIREMENTS FOR TAR.

	Grade RT-1	Grade RT-2	Grade RT-3	Grade RT-4	Grade RT-5	Grade RT-6	Grade RT-7	Grade RT-8	Grade RT-9	Grade RT-10	Grade RT-11	Grade RT-12	Grade RT.C.B. -5	Grade RT.C.B. -6
Water by volume, max., per cent	2.00	2.00	2.00	2.00	1.5	1.5	1.0	none	none	none	none	none	1.0	1.0
Specific gravity at 25/25 C. (77/77 F.), min.	1.08	1.08	1.09	1.09	1.10	1.10	1.12	1.14	1.14	1.15	1.16	1.16	1.09	1.09
Specific viscosity,[a] Engler, 50 cc... at 40 C. (104 F.)	5 to 8	8 to 13	13 to 22	22 to 35	.....	.....	.....	.....	.....	.....	.....	.....	17 to 26	26 to 40
Specific viscosity,[a] Engler, 50 cc... at 50 C. (122 F.)	.....	.....	.....	.....	17 to 26	26 to 40	.....	.....	.....	.....	.....	.....	.....	.....
Float test,[a] sec... at 32 C. (89.6 F.)	.....	.....	.....	.....	.....	.....	50 to 80	80 to 120	120 to 200	.....	.....	.....	.....	.....
Float test,[a] sec... at 50 C. (122 F.)	.....	.....	.....	.....	.....	.....	.....	.....	.....	75 to 100	100 to 150	150 to 220	.....	.....
Distillation test on water-free material: Total distillate by weight, per cent: to 170 C. (338 F.)	7.0 max.	7.0 max.	7.0 max.	5.0 max.	5.0 max.	5.0 max.	3.0 max.	1.0 max.	1.0 max.	1.0 max.	1.0 max.	1.0 max.	2.0 to 8.0	2.0 to 8.0
Distillation test on water-free material: to 200 C. (392 F.)	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	5.0 min.	5.0 min.
Distillation test on water-free material: to 235 C. (455 F.)	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	8.0 to 18.0	8.0 to 18.0
Distillation test on water-free material: to 270 C. (518 F.)	35.0 max.	35.0 max.	30.0 max.	30.0 max.	25.0 max.	25.0 max.	20.0 max.	15.0 max.	15.0 max.	10.0 max.	10.0 max.	10.0 max.	.....	.....
Distillation test on water-free material: to 300 C. (572 F.)	45.0 max.	45.0 max.	40.0 max.	40.0 max.	35.0 max.	35.0 max.	30.0 max.	25.0 max.	25.0 max.	20.0 max.	20.0 max.	20.0 max.	35.0 max.	35.0 max.
Softening point (ring-and-ball method) of residue from distillation test	30 to 60 C. (86 to 140 F.)	30 to 60 C. (86 to 140 F.)	35 to 65 C. (95 to 149 F.)	35 to 65 C. (95 to 149 F.)	35 to 70 C. (95 to 158 F.)	35 to 70 C. (95 to 158 F.)	35 to 70 C. (95 to 158 F.)	35 to 70 C. (95 to 158 F.)	35 to 70 C. (95 to 158 F.)	40 to 70 C. (104 to 158 F.)	40 to 70 C. (104 to 158 F.)	40 to 70 C. (104 to 158 F.)	40 to 70 C. (104 to 158 F.)	40 to 70 C. (104 to 158 F.)
Sulfonation index (when specified): Total distillate: To 300 C. (572 F.)	8 max.	7 max.	6 max.	6 max.	5 max.	5 max.	.....	.....	.....	.....	.....	.....	.....	.....
Sulfonation index (when specified): Total distillate: 300 to 355 C. (572 to 671 F.)	1.5 max.	1.5 max.	1.5 max.	1.5 max.	1.5 max.	1.5 max.	.....	.....	.....	.....	.....	.....	.....	.....
Total bitumen (soluble in carbon disulfide) by weight, min., per cent	88	88	88	88	83	83	78	78	78	75	75	75	80	80

[a] The consistency limits are subdivided into grades RT-1 to RT-12, inclusive, and grades RT.C.B.-5, RT.C.B.-6, so that material may be chosen to meet the local conditions temperature, road conditions, and climate.

Float Test for Bituminous Materials (A.S.T.M. Designation: D 139).[3]

(*f*) *Distillation.*—Standard Method of Test for Distillation of Tar Products Suitable for Road Treatment (A.S.T.M. Designation: D 20).[3]

(*g*) *Softening Point.*—Standard Method of Test for Softening Point of Bituminous Materials (Ring-and-Ball Method) (A.S.T.M. Designation: D 36).[3]

(*h*) *Sulfonation Index.*—Standard Method of Test for Sulfonation Index of Road Tar (Method T 108)[5] of the American Association of State Highway Officials.

(*i*) *Total Bitumen.*—Standard Method of Test for Determination of Bitumen (A.S.T.M. Designation: D 4).[3]

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[5] See "Standard Specifications for Highway Materials and Methods of Sampling and Testing," the Am. Assn. State Highway Officials, Part II, p. 116 (1942).

AMERICAN ASSOCIATION STATE
HIGHWAY OFFICIALS STANDARD
A.A.S.H.O. NO.: T 40-42

Tentative Methods of
SAMPLING BITUMINOUS MATERIALS[1]

A.S.T.M. Designation: D 140 - 41 T

ISSUED, 1939; REVISED, 1941.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

General Directions

1. Samples may be taken for either of two purposes:

(*a*) To represent as nearly as possible an average of the bulk of the material sampled.

(*b*) To ascertain the maximum variation in characteristics which the material may possess. In either case they shall be obtained by methods hereinafter described.

Protection and Preservation

2. (*a*) Care shall be taken that the samples are not contaminated with dirt or any other extraneous matter and that the sample containers are perfectly clean and dry before filling.

(*b*) Immediately after filling, the sample containers shall be tightly closed and properly marked for identification on the container itself or on a linen tag attached to the container.

Time and Place

3. (*a*) Whenever practical, bituminous materials shall be sampled at the point of manufacture, and at such time as to allow the tests, controlling acceptance or rejection, to be made in advance of shipment.

(*b*) When impracticable to take samples at the point of manufacture, they should be taken from the shipment immediately upon delivery.

Size of Samples

4. For routine laboratory examination of acceptability of a given lot, not less than 1 qt. of material should be submitted which should be representative of the average sample collected as hereinafter described.

Containers

5. Containers for liquid bituminous materials shall be small-mouth cans with cork-lined screw caps. Containers for semisolid and solid materials shall be friction top cans.

[1] Under the standardization procedure of the Society, these methods are under the joint jurisdiction of A.S.T.M. Committee D-4 on Road and Paving Materials and Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] Prior to their present publication as tentative, these methods were published as tentative from 1922 to 1925, being revised in 1923. They were adopted in 1925, published as standard from 1925 to 1939, but withdrawn and republished as tentative in 1939.

SAMPLING AT PLACE OF MANUFACTURE

From Bulk Storage

6. The inlet and outlet to the storage tank shall be sealed and a 1-gal. sample drawn from the top, middle, and bottom contents. The sample may be taken from drain cocks on the side of the tank, if such are available. Enough material should be discarded to insure a representative sample. Otherwise, samples may be taken by lowering, weighted bottles or cans into the material. The bottle or can should be fitted with a stopper which can be removed by a string or wire attached to it after it has been lowered to the proper depth. The three samples from bulk storage shall be tested separately for consistency in order to detect stratification. They may then be combined and thoroughly mixed for other tests that may be required.

During Loading

7. Where tank cars, distributors, or barrels are being filled, samples may conveniently be taken from the pipe line through which the material is flowing, as hereinafter described.

A. When Material is Pumped Under Pressure

Material Under Pressure

8. (*a*) The sampling pipe shall be inserted into a rising section of the pipe line on the discharge side of the pump. The sampling pipe shall be not more than one-eighth the diameter of the line pipe and its opening should be turned at an angle of 90 deg. facing the flow of the liquid. This pipe shall be provided with a plug cock and shall discharge into a receiving drum of 50-gal. capacity. The plug cock shall be so adjusted that there is a steady continuous flow of bituminous material through it and shall be so regulated that the receiver will fill in the same time that is required to make the entire pumping. In the case of semisolid materials, the receiver shall be provided with a steam coil which shall keep the contents at a temperature just above the liquefying point. At completion of the pumping, the receiver shall be thoroughly agitated, and a 1-qt. sample taken therefrom. The sampling shall be so regulated that for each 1000 gal. of material pumped, at least 1 gal. of sample is taken; but 40 gal. shall be the limit required for any one cargo.

(*b*) Care should be taken that the drip cock, after once having been set, allows a constant flow during the pumping.

(*c*) This method is also applicable to gravity flows where the pipe line is completely filled by the outflowing liquid and has a rising section.

B. When Materials Flow by Gravity

Material Flowing by Gravity

9. Materials flowing by gravity through pipes which are not completely filled may be sampled by taking dipperfuls at the outlet at frequent and regular intervals. These samples shall be combined and the total sample shall be not less than 0.1 per cent of the whole material. The samples shall be collected in a receiver and resampled as described in Section 8.

SAMPLING AT POINT OF DELIVERY

Tank Cars

10. (*a*) The material in the car should be agitated or otherwise mixed prior to sampling, if possible. Liquid bituminous materials shall be sampled before heating, if possible, but if heating is necessary at no time shall the temperature exceed the application temperature. Semisolid or solid bituminous material shall be rendered fluid by heat-

ing. A careful observation of the condition of the material in regard to the presence of free water on top of the material in the car shall be made.

(*b*) Samples may be taken by means of a weighted bottle or can as described in Section 6 from the top, middle, and botton of the material in the car or from the unloading pipe line as described in Section 7. The sample bottle shall be dried and any extraneous material on the outside removed before pouring the sample into the shipping container. A suitable sampling device is shown in Fig. 1. Tests on the individual samples will give a better indication of the variations which occur in the material, but the samples should be combined to form a composite sample when it is desired to determine the average condition.

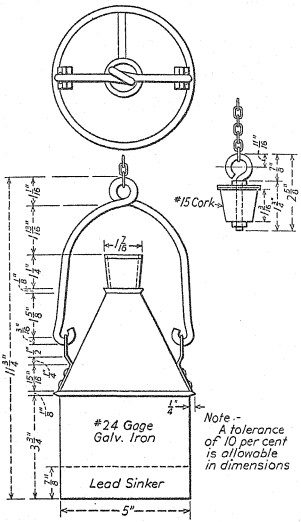


FIG. 1.—Bottle Sampler for Tank Cars.

Distributors

11. Samples may be taken from distributors by means of a weighted bottle or can, as described in Section 6. One sample shall be sufficient.

Barrels, Drums, and Cakes

12. (*a*) *Semisolid or Solid Materials.*— Where the lot of material to be sampled is obviously from a single run or batch of the producer, one package or cake shall be selected at random and sampled as described in the following paragraph. Where the lot of material to be sampled is not obviously from a single run or batch of the producer, or where the single sample selected as described above fails on test to conform to the requirements of the specifications, a number of packages or cakes shall be selected at random equivalent to the cube root of the total number of packages or cakes in the lot. For convenience, the following table is given, showing the number of samples to be selected for shipments of various sizes:

Packages or Cakes in Shipment	Packages or Cakes Selected
2 to 8	2
9 to 27	3
28 to 64	4
65 to 125	5
126 to 216	6
217 to 343	7
344 to 512	8
513 to 729	9
730 to 1000	10
1001 to 1331	11

Samples shall be taken from at least 3 in. below the surface and at least 3 in. from the side of the container or cake or from the center of a cake. A clean hatchet may be used if the material is hard enough to shatter and a broad stiff putty knife if the material is soft. An auger, or brace and $\frac{3}{4}$-in. bit, or other suitable means may also be used.

When more than one package or cake in a lot is sampled, each individual sample shall be not less than ¼ lb. in weight. When the lot of material is obviously from a single run or batch of the producer, all samples from the lot shall be melted and thoroughly mixed, and an average sample taken from the combined material for examination. In case more than a single run or batch of the producer is present and the batches can be clearly differentiated, a composite sample shall be prepared for examination from each batch. Where it is not possible to differentiate between the various batches each sample shall be examined separately.

(*b*) *Liquid Materials.*—Samples of liquid bituminous materials from barrels or drums shall be secured by taking 1 qt. of material from packages selected at random according to the cube root method described in Paragraph (*a*). When the lot of material is obviously from a single run or batch of the producer the samples shall be combined and thoroughly mixed and an average sample taken from the combined material. In case more than one run or batch is present and can be clearly differentiated, a composite sample shall be prepared from each batch. Where it is impossible to differentiate between the various batches or runs, each sample shall be examined separately.

Bulk and Bags

13. Solid bituminous materials in crushed fragments or powder, such as gilsonite, grahamite, etc., shall be sampled in accordance with the Standard Method of Sampling Coal for Analysis (A.S.T.M. Designation: D 21) of the American Society for Testing Materials.[3]

[3] 1944 Book of A.S.T.M. Standards, Part III.

AMERICAN ASSOCIATION STATE
HIGHWAY OFFICIALS STANDARD
A.A.S.H.O. NO.: T 47-42

Tentative Method of Test for

LOSS ON HEATING OF OIL AND ASPHALTIC COMPOUNDS[1]

A.S.T.M. Designation: D 6 – 39 T

ISSUED, 1939; REVISED, 1939.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. This method of test is intended for the determination of the loss in weight (exclusive of water) of oil and asphaltic compounds when heated as hereinafter prescribed.

Apparatus

2. (*a*) *Oven.*—The oven shall be rectangular in form with double walls and heated by electricity. Its interior dimensions shall be as follows: Height, exclusive of space occupied by the heating element, not less than 29.21 cm. (11.5 in.); width and depth, not less than 29.85 cm. (11.75 in.). The oven shall have in front a tightly-fitting hinged door, which shall provide a clear opening substantially of the interior height and width of the oven. The door may contain a window with linear dimensions of at least 10 cm. (4 in.), and with two sheets of glass separated by an air space, through which a vertical thermometer located as specified in Section 4 may be read without opening the door; or the oven may be provided with an inner glass door, through which the thermometer may be observed on opening the outer door momentarily.

The oven shall be adequately ventilated by convection currents of air, and for this purpose the oven shall be provided with openings for the entrance of air and the exit of heated air and vapors. Openings for the entrance of air in interior walls of the oven shall be symmetrically arranged in the bottom or in side walls near the bottom, and shall be so placed that incoming air will circulate around the heating elements; the openings shall have a total area of not less than 1.3 sq. cm. (0.2 sq. in.). Openings for the exit of heated air and vapors in interior walls of the oven shall be symmetrically arranged in the top or in side walls near the top, and shall have a total area of not less than 1.3 sq. cm. (0.2 sq. in.) nor more than 12.9 sq. cm. (2.0 sq. in.).

The oven shall be provided with a perforated metal circular shelf approximately 24.77 cm. (9.75 in.) in diameter.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] Prior to its present publication as tentative, this method was published as standard from 1911 to 1939, being revised in 1916, 1920, 1927, 1930, 1933, but withdrawn, revised, and republished as tentative in 1939.

Revision accepted by Committee E-10 on Standards, August 24, 1939. Editorially revised in 1941.

A recommended form of aluminum shelf is shown in Fig. 1. This shelf shall be placed in the center of the oven with respect to all dimensions of the interior of the oven, shall be suspended by a vertical shaft and provided with mechanical means for rotating it at the rate of 5 to 6 rpm.

(*b*) *Thermometer.*—An A.S.T.M. Loss on Heat Thermometer graduated in Centigrade degrees, having a range of 155 to 170 C., and conforming to the requirements for this thermometer as prescribed in the Standard Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1) of the American Society for Testing Materials.[3]

(*c*) *Container.*—The container in which the sample is to be tested shall be of metal or glass, cylindrical in shape, and shall have a flat bottom. Its inside dimensions shall be substantially as follows: diameter, 55 mm. (2.17 in.), depth, 35 mm. (1.38 in.).

NOTE.—Containers known in the drug trade as seamless "ointment boxes" may be obtained in dimensions conforming to the above requirements.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

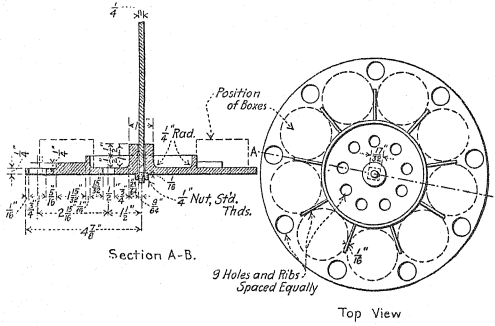


FIG. 1.—Aluminum Shelf.

Preparation of Sample

3. (*a*) The sample as received shall be thoroughly stirred and agitated, warming, if necessary, to insure a complete mixture before the portion for analysis is removed.

(*b*) The material under examination shall first be tested for water and if water is found to be present, it shall be removed by suitable methods of dehydration before the material is subjected to the loss on heating test; or another sample shall be obtained which is free from water.

Procedure

4. (*a*) A 50.0 ± 0.5 g. sample of the water-free material to be tested shall be placed in a tared container conforming to the requirements of Section 2 (*c*) and weighed to the nearest 0.01 g. If the material has been heated to facilitate transfer, the sample shall be cooled to room temperature before making this initial weighing. The oven shall be brought to a temperature of 163 C. (325 F.), and the container with the weighed sample placed on and near the circumference of the circular shelf, in one of the recesses if the rec-

ommended shelf is used. The oven shall then be closed and the shelf rotated during the entire test at a rate of 5 to 6 rpm. The temperature shall be maintained at 163 ± 1 C. (325 ± 1.8 F.) for 5 hr. after the sample has been introduced and the oven has again reached that temperature. The 5-hr. period shall start when the temperature reaches 162 C., and in no case shall the total time that a sample is in the oven be more than 5 hr. and 15 min. At the conclusion of the heating period the sample shall be removed from the oven, cooled to room temperature, weighed to the nearest 0.01 g., and the loss due to heating calculated.

(*b*) Temperatures shall be determined by means of the specified thermometer, which shall be supported from the shaft of the circular shelf in a vertical position approximately 1.9 cm. (0.75 in.) inside the periphery of the shelf, and with the bottom of the thermometer bulb 0.25 in. above the shelf.

NOTE 1.—If additional periods of heating are desired it is recommended that they be made in successive increments of 5 hr. each.

NOTE 2.—When it is required that the penetration or other characteristics of the sample after heating be determined, the residue should be melted in the container at the lowest possible temperature and thoroughly mixed by stirring, taking care to avoid incorporating air bubbles in the material. For the penetration test, the well-mixed residue shall be brought to standard temperature and tested as prescribed in the Standard Method of Test for Penetration of Bituminous Materials (A.S.T.M. Designation: D 5) of the American Society for Testing Materials.[3] For other tests, the well-mixed residue should be tested according to standard conditions as required by the test procedure involved.

Precautions

5. Under ordinary circumstances a number of samples having about the same degree of volatility may be tested at the same time. Samples varying greatly in volatility should be tested separately. When extreme accuracy is required not more than one material should be tested at one time and duplicate samples of it should be placed simultaneously in the oven. Such duplicates shall check within the limits of accuracy given in Section 6. Results obtained on samples showing evidences of foaming during the test shall be rejected.

Reproducibility of Results

6. Up to 5 per cent loss in weight the results obtained may be considered as correct within 0.5. Above 5 per cent loss in weight the numerical limit of error increases 0.01 for every 0.5 per cent increase in loss by volatilization as follows:

Volatilization Loss, per cent	Numerical Correction	True Volatilization Loss, per cent
5.0	±0.50	4.50 to 5.50
5.5	±0.51	4.99 to 6.01
6.0	±0.52	5.48 to 6.52
10.0	±0.60	9.40 to 10.60
15.0	±0.70	14.30 to 15.70
25.0	±0.90	24.10 to 25.90
40.0	±1.20	38.80 to 41.20

Tentative Method of Test for

HOT EXTRACTION OF ASPHALTIC MATERIALS AND RECOVERY OF BITUMEN BY THE MODIFIED ABSON PROCEDURE[1]

A.S.T.M. Designation: D 762 - 44 T

ISSUED, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. This method of test covers the procedure for the extraction of benzol soluble bitumen from asphaltic mixtures, the removal of mineral matter from the solution and the recovery of the bitumen from solution in sufficient quantity for further testing.

NOTE.—This method has been studied by the committee for asphalts harder than 150 penetration. Suitability of the method for softer residues has not been determined.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Oven.*—An oven capable of maintaining the temperature at 210 to 220 F.

(*b*) *Balance.*—A balance capable of weighing 5000-g.

(*c*) *Extractor.*—An extraction apparatus as shown in Fig. 1.

(*d*) *Centrifuge.*—A centrifuge capable of handling two 8-oz. wide-mouth bottles at 770 times gravity.[3]

(*e*) *Bottles.*—A supply of 8-oz. wide-mouth bottles.

(*f*) *Distillation Assembly.*—A distillation assembly as shown in Fig. 3 and consisting of the following items:

(*1*) A heat-resistant[4] glass distillation column 250 mm. in length and 25 mm. in diameter, and provided with a side arm 150 mm. in length and 8 mm. in diameter.

(*2*) An iron tripod.

(*3*) A 6 by 6-in. 20-mesh wire gauze with an asbestos center.

(*4*) A gas burner.

(*5*) A water-jacketed condenser 475 to 500 mm. in length.

(*6*) Three thermometers conforming to the requirements for thermometer E 1 (7 F - 39) as prescribed in the Standard Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1).[5]

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] Accepted by the Society at annual meeting, June, 1944.

[3] An "International" No. 2 Centrifuge at 1900 rpm. will meet these requirements.

[4] Pyrex glass is satisfactory for this purpose.

[5] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

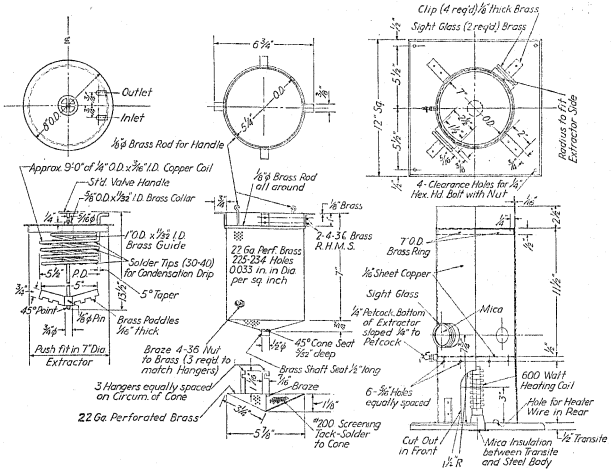


FIG. 1.—Extraction Assembly.

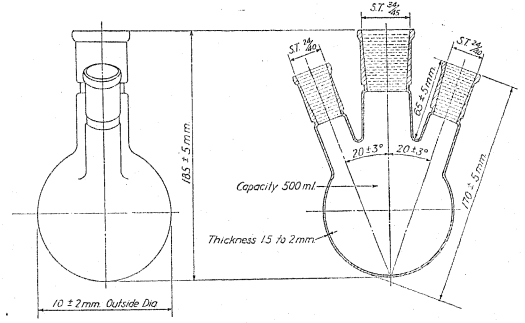


FIG. 2.—Distillation Flask.

(*7*) A 250-ml. Erlenmeyer receiving flask and a 250-ml. Erlenmeyer filtering flask.

(*8*) A 500-ml. graduated cylinder.

(*9*) Corks of assorted sizes.

(*10*) A ringstand and supports.

(*11*) A distillation flask as shown in Fig. 2.

(*12*) An oil bath for the distillation flask.

Sample

4. A sample of sufficient size to result in at least 100 g. of recovered bitumen is required. About 1000 g. of sheet-asphalt mixtures will usually be sufficient unless the largest particles in the sample are 1 in., in which case 2000 g. will usually be required. Mixtures containing larger aggregates will require still larger samples.

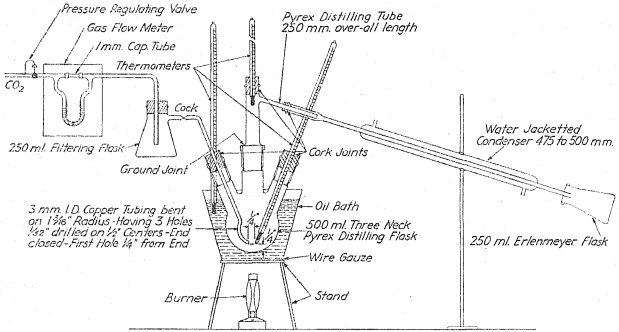


FIG. 3.—Distillation Assembly.

(*13*) A gas flow meter.

(*14*) A gas inlet coil.

(*15*) A cylindrical, flat-bottom, seamless tin container of 6-oz. capacity. The container shall be 70 mm. ($2\frac{3}{4}$ in.) in diameter and 45 mm. ($1\frac{3}{4}$ in.) in depth (Note).

NOTE.—Containers known in the drug trade as seamless "ointment boxes" may be obtained in the dimensions conforming to these requirements.

Reagents

3. (*a*) *Benzol* (thiopene free, dry, with a boiling range of 79.5 to 81.5 C.).

(*b*) *Carbon dioxide.*

Procedure

5. (*a*) The sample shall be placed in the oven at 210 to 220 F. for 15 min., broken into pieces and dried in the oven for an additional 30 min. The desired amount of the sample shall be weighed to the nearest 5 g. and placed in the basket of the extractor with the stirrer in place. The extractor shall be charged with 400 ml. of benzol and the wire cone hung on the bottom of the basket. The basket shall be inserted in the extractor, the condenser, cover placed on the extractor and the handle placed on the stirrer. Cold water shall be cir-

culated through the condenser. The electric heater shall be connected and the sample extracted until the benzol is colorless (Note). When the sample contains a mineral filler, the stirrer shall be turned by hand one-half turn every 15 min. (after the benzol becomes straw colored) to break up the settled filler and remove the last traces of bitumen.

NOTE.—This can be observed by placing a light at one window of the extractor and observing the dripping benzol through the other window.

(*b*) The benzol solution shall be drawn off and the volume increased to 400 ml. by the addition of fresh benzol, using this solution to wash the extractor if necessary. The solution shall be poured into two 8-oz. wide-mouth bottles, balanced accurately, stoppered, and placed in the centrifuge. The solution shall be centrifuged at room temperature for 30 min. at 770 times gravity using the distance (in feet) from the center of the centrifuge to a point midway in the liquid, as the value of R in the following formula for calculation of speed of the centrifuge:

$$\text{Speed, rpm.} = \frac{1500}{\sqrt{R}}$$

(*c*) The solution shall be poured into a previously weighed 500-ml., three-neck flask, care being taken not to disturb or include the sediment.

(*d*) Using the distillation assembly shown in Fig. 3, the temperature shall be raised inside the flask to 300 F. (148.9 C.) at such a rate that the benzol is collected at a rate of 50 to 70 drops per min. As soon as this rate falls off, carbon dioxide gas shall be admitted slowly, increasing to a rate of 800 to 900 ml. per min. The contents of the flask shall be maintained at 295 to 305 F. (146.1 to 151.7 C.) for exactly 30 min. with full carbon dioxide gas rate. The outside bath temperature ordinarily shall be held 15 to 25 F. (8 to 14 C.) higher than the inside in order to maintain a sample temperature of 295 to 305 F. (146.1 to 151.7 C.).

(*e*) The flame shall be removed, the carbon dioxide gas shut off, and the apparatus disassembled. The outside of the flask shall be cleaned and the flask and contents weighed. The percentage of bitumen shall be determined by the following formula:

$$\text{Bitumen, per cent} = \frac{\text{wt. of flask and contents} - \text{wt. of flask}}{\text{wt. of original sample}} \times 100$$

(*f*) The contents of the flask shall then be poured into the 6-oz. container and cooled.

Tentative Methods of

SURVEYING AND SAMPLING SOILS FOR HIGHWAY SUBGRADES[1]

A.S.T.M. Designation: D 420 - 42 T

ISSUED, 1935; REVISED, 1940, 1942.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Purpose of Subgrade Survey

1. The purpose of the subgrade survey is to furnish the engineer with significant information on the following subjects:

(*a*) The final location of the road both vertically and horizontally.

(*b*) The selection of suitable fill material.

(*c*) The design of the roadway section.

(*d*) The design and location of ditches, culverts, and drains.

(*e*) The need for subgrade treatment and the type of treatment required.

(*f*) The selection of the type of road surface and its design.

Scope of Subgrade Survey

2. These methods cover procedures for the surveying and sampling, for identification and subgrade profile purposes, of soils for highway subgrades but do not include the examination of soils for their supporting value as foundation materials or stability in slopes. These same methods can be applied to surveying subgrades for airport runways, parking, or taxiway areas.

NOTE.—Investigation of soils under structures or fills for their supporting value and the stability of slopes in embankments or cuts should not be overlooked by the engineer and where necessary a thorough study of these factors should be made. This will require special procedures and equipment not covered in these methods.

Equipment for Subgrade Survey

3. The following equipment is required to make a subgrade survey:

(*a*) One 3-ft. soil auger and three 3-ft. extensions as illustrated in Fig. 1.

(*b*) Two small pipe wrenches.

(*c*) One light pick.

(*d*) One shovel.

(*e*) A supply of sample bags.

(*f*) A supply of tags for marking samples.

(*g*) A ball of twine.

(*h*) One engineer's level.

(*i*) One hand level.

(*j*) One 12-ft. level rod, three-section.

(*k*) One 100-ft. metallic tape.

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-18 on Soils for Engineering Purposes.

[2] Latest revision accepted by Committee E-10 on Standards, August 24, 1942.

(*l*) One 12 by 15 in. strip of stiff cardboard.

(*m*) One roll of 20-in. cross-section paper, 10 divisions to the inch each way.

(*n*) Notebooks.

(*o*) A supply of survey stakes.

(*p*) One camera and supply of films.

(*q*) A supply of keel.

Soil Profile Determination

4. The soil profile shall be determined by examining the soil in its natural field condition. This work may be accomplished by examining excavations, road cuts, etc.; however, the most commonly used method is, of necessity, the soil auger. There is no definite rule to follow in making these examinations, except that the soil should be examined at intervals close enough to determine the soil type and by borings deep enough to penetrate the more or less nonuniform layers of soil or soil material.

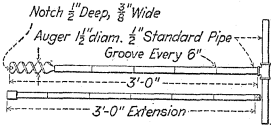


FIG. 1.—Auger for Obtaining Soil Samples.

Examination of Soil Section

5. The examination of the soil section should proceed in a systematic way, paying particular attention to the properties described in Sections 6 to 12:

6. *Texture.*—The successive layers or horizons differing in texture, or in fineness or coarseness of the material, should be carefully examined. The examination should extend to a depth of at least 5 or 6 ft. The texture of each layer and its thickness should be described.

7. *Color.*—The successive layers which can be differentiated according to differences in color should be noted. Each layer should be described and its thickness given.

8. *Structure.*—The several layers that differ according to structure should be examined carefully, structure being defined as the kind and size of soil particle aggregation. Special note should be made of horizons with the following:

(*a*) Fine granular structure (granules about the size of bird shot or smaller).

(*b*) Coarse granular structure (granules ranging up to ½ in. or more in diameter and usually more angular or irregular in shape than the granules making up the fine granular structure).

(*c*) Layered or platey structure, in which the material splits into thin plates (not to be confused with stratification).

(*d*) Buckshot structure, in which the soil on drying breaks up into angular fragments (found to characterize heavy clays usually having a considerable percentage of lime).

(*e*) Single-grain structure in which the material is like flour or sand with no aggregation of particles.

9. *Consistency.*—A determination should be made of the successive layers or horizons differing in consistency (stickiness, friability, plasticity). A description of each should be given and its thickness noted.

10. *Compactness.*—The relative compactness of the several layers should be determined, as measured by the degree of resistance to the penetration of a pointed instrument.

11. *Cementation.*—It should be determined whether or not resistance to penetration is due in any horizon to cementation. If so, the probable cementing material (light colored or reddish, very strongly cemented or weakly cemented) should be ascertained.

12. *Chemical Composition.*—(*a*) While the determination of the chemical composition of the various parts of the soil

section or profile can not be performed in the field by the usual field methods, there are certain features that may be determined in at least a qualitative way. Field examination can detect the presence of horizons with concentrations of organic matter or of salts of the alkalies and alkaline earths.

(*b*) The organic matter referred to in Paragraph (*a*) is that contained in the soil and not that lying on the soil. Of this there are two kinds to be looked for: namely,

(*1*) The organic matter in the surface soil is recognized by the dark color, and the approximate relative percentage present is indicated by the intensity of the dark color. The determination of the thickness of the dark-colored layer in the virgin soil is very important.

(*2*) In some soils, usually confined to regions with a cool, moist climate, there is present, at a depth ranging from 6 in. or less to somewhat more than a foot, a layer of brown or coffee-brown organic matter forming a layer 6 or 8 in. in thickness.

(*c*) The salts of the alkalies and alkaline earths accumulate in the soil under favorable conditions. Alkaline deposits are commonly found in semiarid and desert regions where the rainfall is insufficient to remove these salts from the soil. In these areas the location and extent of all such deposits shall be carefully noted.

(*d*) Horizons of lime carbonate accumulation may be readily identified and should be looked for where the rainfall is less than 17 to 18 in. per year in cool to cold climates and 30 in. per year in hot or very warm climates. The unweathered material beneath the soil in any region, arid, subhumid, or humid may have a high percentage of lime carbonate, but such material should not be confused with the horizon of true lime carbonate accumulation.

(*e*) Sesquioxides accumulate in the soil under favorable conditions. Since accumulations of aluminum hydroxide are not readily identified by the usual field methods these may be left out of consideration. Accumulations of iron oxides are, however, important to consider.

(*f*) Iron oxides occur in two forms: first form consists of accumulations of finely divided or colloidal iron oxide (hydroxide). The degree of concentration may be determined, within a rather wide range of error, by the intensity of the red color. The existence of red horizons in the soil profile should be noted and should be illustrated with samples, even though they be small. The second type consists of accumulations of iron oxide concretions or large masses, usually porous or slaglike. This statement does not refer to iron-stone slabs or ferruginous sandstone layers which may be found in many places in the parent geological formations. In hot countries the accumulations referred to here take the form of thick masses of porous slaglike iron oxide lying at depths ranging from somewhat less than 3 to more than 15 ft. They may consist of fragments scattered over the surface.

Selection of Samples

13. A 5-lb. sample of soil from each layer shall be obtained with pick and shovel from the exposed back slopes or from test pits dug at locations indicated by the borings. Each sample shall be placed in a canvas bag, tied securely, marked with proper identification, and shipped to the laboratory. A sufficient number of samples shall be taken to determine the range in test results for what appears to be the same layer.

Mapping the Soil Profile

14. The detailed mapping of soil profiles shall be accomplished in the following manner:

(*a*) Vertical soil sections shall be examined at frequent intervals and clas-

sified into layers according to the method described above.

(*b*) The limits of the various layers shall be plotted as shown in Fig. 2 (*a*), alternate numbers being used to indicate the layers. Thus at test hole No. 1, the consecutive layers are numbered 1, 3, 5, etc., so as to allow the inclusion of any other layer which might enter the profile within the section mapped. At test hole No. 4, layer 2 is mapped between layers 1 and 3. This requires the examination of another test hole (No. 5), or perhaps several, between Nos. 3 and 4, to determine the horizontal limits of layer 2.

(*c*) The profile shall be completed by connecting the points marking the limits of the layers as shown in Fig. 2 (*b*).

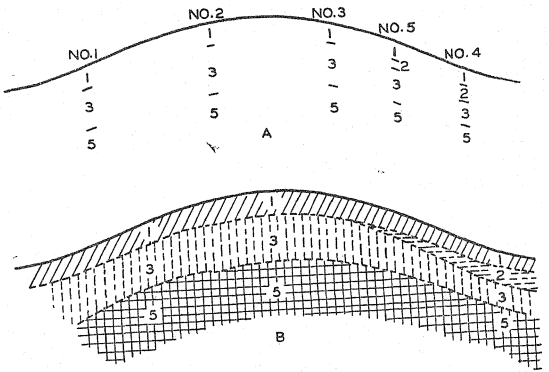


FIG. 2.—Example of Soil Profile Map.
Scales (approximate due to reductions in reproduction):
Horizontal.....$\frac{1}{2}$ in. = 50 ft.
Vertical.....$\frac{1}{2}$ in. = 5 ft.

SUBGRADE SURVEY TO OBTAIN INFORMATION REGARDING ROADS IN SERVICE

Subgrade Survey

15 The section for study having been selected, the survey shall be carried out as follows:

(*a*) The section shall be staked out, the original construction stations being used if possible. Arbitrary stations will serve the purpose when it is not convenient to locate original stations.

(*b*) Cross-sections shall be taken every 50 ft. along the center line or oftener if topography requires, and for a distance 150 ft. on each side of the center line. Elevations shall be obtained with an engineer's level to the nearest 0.1 ft. The accuracy of an engineer's level is necessary for the construction of center-line and bank-line profiles, but a hand level is sufficiently accurate for the topography adjacent to the highway. An assumed elevation may be used as a bench mark.

(*c*) A plan of the roadway shall be drawn to a scale of 50 ft. to the inch, showing the type of pavement, type of failure, portion of roadway which is built over an older road, if any, and any special construction.

(*d*) Cross-section notes shall be plotted to the same scale as the plan of roadway and contours drawn in by interpolation.

(*e*) The bank-line profiles shall be drawn on the cross-section paper using a horizontal scale of 50 ft. to the inch and a vertical scale of 5 ft. to the inch. The center line profile and the grade line of the pre-existing road, if any, shall be projected upon the bank-line profiles.

Examining and Mapping Soil Profile

16. The soils shall be mapped and the profiles plotted according to the procedure outlined in Section 14. The desired information may be conveniently obtained and recorded in the following manner:

(*a*) Scrape down the back slopes so that the original undisturbed material is exposed and plot the limits of the various layers on the prepared profile sheets. Supplement this work by soil auger borings so that a profile is obtained to a depth of at least 3 to 5 ft. below the center-line grade. The depth will vary with the uniformity of the soil layers or soil material. Any variations in moisture content should be specially noted. Obtain the elevations of the limits of the different layers in the exposed back slopes by means of a hand level, the elevation of the center line being used as a bench mark.

(*b*) In a separate notebook describe each layer in detail according to the properties enumerated in Sections 6 to 12, inclusive, recording also the relative imperviousness or porosity of the individual layers.

(*c*) Examine the soil every 50 ft. or less, depending on the uniformity of the profile (see Section 14).

(*d*) On the plan of the roadway plot the limits of the various soil layers found directly under the surfacing material. When the roadway is cut through uniform layers of soil material, the limits are obtained by constructing cross-sections from the bank-line profiles. When the roadway is constructed of fill material or cut through a heterogeneous soil material, the limits are determined by soil auger borings along the centerline of the roadway; or if a permanent pavement is in place by boring or trenching immediately adjacent to the pavement edge.

Selection of Samples

17. Adequate samples shall be secured as described in Section 13.

Photographs of Road Surfaces

18. Photographs shall be taken illustrating the condition of the pavement, the shoulders, the back slopes, the ditches, and the appearance of the soil layers.

Analysis of Data

19. The data collected in the manner described above shall be analyzed, together with the laboratory test results, and information on the following subjects developed:

(*a*) The relation that exists between the pavement condition, the field characteristics of the soil, and the physical properties of the soil as determined in the laboratory.

(*b*) The possible reasons for pavement failure.

(*c*) Possible curative measures for the case under examination.

(*d*) Preventive measures which may be applied in the future.

SUBGRADE SURVEY TO OBTAIN INFORMATION WITH RESPECT TO THE DESIGN OF A NEW ROAD

Study of Soil Information and Preliminary Survey

20. Before starting a subgrade survey

of this kind the engineer should make a study of all the existing information on the soil types in that vicinity. Wherever soil maps prepared by the Soil Survey Section of the Bureau of Plant Industry, U. S. Department of Agriculture, are available they should be carefully studied and the limits of the various soil types and their characteristics should be noted. It must be kept in mind that the detail to which such maps are carried is not particularly adapted to a subgrade survey. Nevertheless, they give a clear idea of the variations which will be encountered. Where this information is not available, a reconnaissance survey should be made of the soil materials on existing highways which parallel the new highway, noting the changes in soil as shown in exposed cuts. The notes should include a complete description of each soil type. The value of the information obtained from this rough survey lies in the fact that similar soil conditions may be expected to accompany similar topographic features.

Subgrade Survey

21. After this information has been digested the survey shall proceed in accordance with the following Sections 22 to 26. The profile of the ground line and the proposed grade line shall be constructed on the same type of sheet as was specified for subgrade surveys of existing roads in service as described in Sections 15 and 16.

Mapping and Sampling Soil Profile

22. Borings shall be made with a soil auger at frequent intervals and each soil type classified into layers, as described in Sections 4 to 14.

(*a*) *Spacing of Borings.*—The spacing of borings should vary with the uniformity of the profile and the topography. A convenient interval, such as the even stations, may be assumed at the beginning. This interval may be varied under the following conditions: (*1*) If the profile is uniform, the interval may be increased; (*2*) When the character of the profile changes, intermediate borings should be made until it is clear that all variations have been mapped; (*3*) Where topography is rolling and grade changes rapidly from cut to fill, borings are necessary only in the cuts; (*4*) Where the original ground line or old road grade is to be covered with fill material, no examination is necessary except to determine the character of the support. If the fill material is to be obtained from borrow ditches along the road, the soil should be examined to the entire depth of the borrow.

(*b*) *Depth of Borings.*—The borings should generally be carried to a depth of at least 3 ft. below the grade line. The depth may vary in accordance with the following stipulations: (*1*) When the road lies within the uniform layers of the soil profile, the boring should extend down to the first layer below the ditch line which would block percolation, or through a pervious layer which would carry water; (*2*) When fill material is to be borrowed from ditches alongside the road, the boring should extend at least to the estimated depth of borrow; (*3*) In the study of frost action the borings should extend to the mean depth of frost in those soil materials showing a high affinity for frost accumulation and in localities where high water tables prevail.

(*c*) When the located line is over an old road, the soils may be mapped by examining the exposed cuts. This work should be supplemented by borings.

(*d*) A notation shall be made of the direction of surface drainage with respect to the proposed roadway.

Mapping Soil Profile

23. The data obtained from the

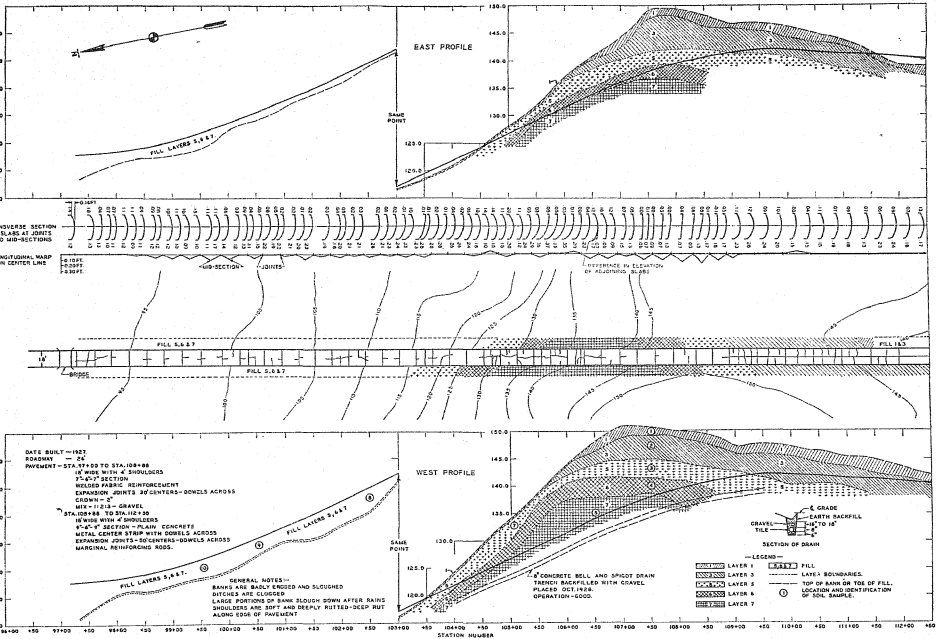


FIG. 3.—Typical Subgrade Survey Sheet for a Road in Service.

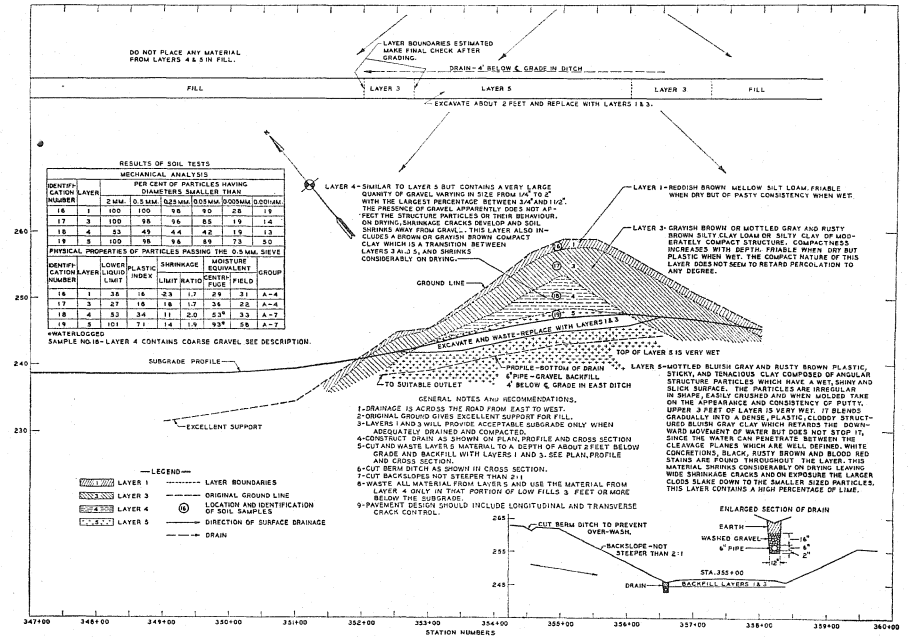


FIG. 4.—Typical Subgrade Survey Sheet for the Design of a New Road.

borings shall be plotted on the prepared profile sheet. On this sheet shall also be indicated the limits of the several types and layers, the relative moisture content at various depths, and the location of culverts and drains.

Examination of Soil Section

24. In a separate notebook the field characteristics of each layer shall be described according to the properties enumerated in Sections 6 to 12, inclusive. The relative imperviousness or porosity of each layer shall also be indicated.

Selection of Samples

25. Adequate samples shall be obtained as described in Section 13.

Analysis of Data

26. (*a*) Recommendations regarding the design of the road surface shall be made on the basis of the known behavior of pavements for which the conditions of soil, climate, and topography are similar.

(*b*) After the road is graded a final check shall be made on the soil as exposed by grading operations.

(*c*) Typical subgrade survey sheets are shown in Figs. 3 and 4.

Tentative Method of Test for

MOISTURE-DENSITY RELATIONS OF SOILS[1]

A.S.T.M. Designation: D 698 - 42 T

ISSUED, 1942.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. This method of test is intended for determining the relationship between the moisture content of soils and resulting densities (oven-dry weight per cubic foot) when the soil is compacted in the laboratory as specified herein.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Mold.*—A cylindrical metal mold having a capacity of $\frac{1}{30}$ cu. ft. with an internal diameter of 4.0 in. and a height of approximately 4.6 in., which has a detachable collar assembly approximately $2\frac{1}{2}$ in. in height, to permit preparing compacted specimens of soil-water mixtures 4.0 in. in diameter and approximately 4.6 in. in height which have a volume of $\frac{1}{30}$ cu. ft. The mold and collar shall be fastened to a detachable base. (See Fig. 1.)

(*b*) *Rammer.*—A metal rammer having a 2-in. diameter circular face and weighing 5.5 lb. The rammer shall be equipped with a suitable arrangement to control the specified drop.

(*c*) *Sleeve.*—A closed cylindrical sleeve slightly less than 4.0 in. in diameter, or similar device, for removing compacted specimens from the mold.

(*d*) *Balances.*—A balance or scale of 25-lb. capacity sensitive to 0.01 lb., and a 100-g. capacity balance sensitive to 0.1 g.

(*e*) *Drying Oven.*—A thermostatically controlled drying oven capable of maintaining temperatures of about 110 C. (230 F.) for drying moisture samples.

(*f*) *Straightedge.*—A steel straightedge 12 in. in length.

Sample

3. A 6-lb. sample of soil taken from a portion of the material passing the No. 4 (4760-micron) sieve shall be air-dried to a slightly damp condition.

Procedure

4. (*a*) The 6-lb. sample shall be thoroughly mixed, then compacted in the cylinder (with the extension attached) in three equal layers, each layer receiving 25 blows from the rammer dropping

[1] Under the standardization procedure of the Society, this method is under the joint jurisdiction of A.S.T.M. Committee D-18 on Soils for Engineering Purposes and Committee D-4 on Road and Paving Materials.

[2] Accepted by Committee E-10 on Standards, September 17, 1942.

free from a height of 12 in. above the elevation of each finally compacted layer. During compaction, the mold shall rest on a uniform, rigid foundation weighing 200 lb. or of equivalent rigidity. The blows shall be uniformly distributed over the surface of the layer being compacted. The extension shall then be removed and the compacted soil carefully trimmed even with the top of the cylinder by means of the straightedge, and weighed.

(*b*) The weight of the compacted sample and cylinder, minus the weight of the cylinder, shall then be multiplied by 30 and the result recorded as the wet weight per cubic foot of the compacted soil.

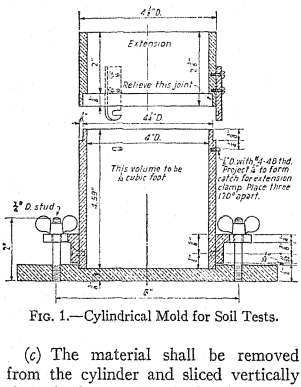


FIG. 1.—Cylindrical Mold for Soil Tests.

(*c*) The material shall be removed from the cylinder and sliced vertically through the center. At least a 100-g. sample shall be taken from the center, weighed immediately, and dried in an oven at 110 C. (230 F.) for at least 12 hr. or to constant weight to determine the moisture content.

(*d*) The remainder of the material shall be broken up until it will pass a No. 4 (4760-micron) sieve. Water in sufficient amounts to increase the moisture content of the soil sample by approximately 1 per cent shall be added and the above procedure repeated for each increment of water added. This series of determinations shall be continued until the soil becomes very wet or there is a substantial decrease in the wet weight of the compacted soil.

Calculations

5. The moisture content and the dry weight of the soil as compacted shall be calculated as follows:

$$M = \frac{A - B}{B - C} \times 100$$

$$W = \frac{W_1}{M + 100} \times 100$$

where:

M = percentage of moisture in the specimen,
A = weight of dish and wet soil,
B = weight of dish and dried soil,
C = weight of dish,
W = dry weight per cubic foot of compacted soil, and
W_1 = wet weight in pounds per cubic foot.

Moisture-Density Relationship

6. (*a*) The calculations in Section 5 shall be made to determine the moisture content and corresponding compacted oven-dry weight (density) of the compacted soil for each test made. The oven-dry weight per cubic foot (density) of the soil shall be plotted as ordinates and corresponding moisture contents as abscissas.

(*b*) *Optimum Moisture Content.*—When the moisture-density relations have been determined for a soil and the results plotted as indicated in Paragraph (*a*), it will be found that by connecting the plotted points with a smooth line, a curve is produced which is, generally,

parabolic in form. The moisture content producing the peak of the curve shall be termed the "optimum moisture content" of the soil under the above compaction.

(*c*) *Maximum Density.*—The oven-dry weight per cubic foot of the soil at "optimum moisture content" shall be termed "maximum density" under the above compaction.

Tentative Method of Test for

CEMENT CONTENT OF SOIL-CEMENT MIXTURES[1]

A.S.T.M. Designation: D 806 – 44 T

ISSUED, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. This method covers a procedure for determining by chemical analysis the cement content of soil-cement mixtures, sampled from a project under construction or after completion.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Analytical Balance.*—An analytical balance with class S weights.

(*b*) *Filter Paper.*—Filter paper including Whatman No. 1, 11 and 15 cm. in diameter; Whatman No. 41, 15 cm. in diameter; and Whatman No. 2, 11 or 15 cm. in diameter.

(*c*) *Miscellaneous Apparatus.*—Supplementary equipment, such as electric ovens, hot plates, a small riffle, a No. 40 (840-micron) sieve with bottom pan and cover, a cast iron mortar and pestle, and a ball mill if possible.

Reagent

3. *Standard Potassium Permanganate Solution (0.1 N).*—Prepare and standardize a 0.1 *N* $KMnO_4$ solution.

NOTE.—The use of a standard 0.1 *N* $KMnO_4$ solution is not necessary when the samples are titrated in accordance with Section 5 (*j*) and the results are calculated in accordance with Section 6 (*b*).

Samples

4. (*a*) Samples of the following shall be selected for the test:

(*1*) *Raw Soil,* representative of the soil phase of the soil-cement mixture,

(*2*) *Cement,* representative of the cement phase of the soil-cement mixture, and

(*3*) *Soil-Cement Mixture* to be analyzed.

(*b*) The gross laboratory sample of each component shall be approximately 200 g. This may be obtained by reducing the field sample in bulk and, if necessary, in particle size through the use of drying, riffling, and grinding processes.

Procedure

5. (*a*) Dry 25 g. of each of the samples in an oven to constant weight at 110 C. (230 F.) to remove free moisture.

[1] Under the standardization procedure of the Society, this method is under the joint jurisdiction of A.S.T.M. Committee D-4 on Road and Paving Materials and Committee D-18 on Soils for Engineering Purposes.

[2] Accepted by Committee E-10 on Standards, August, 28, 1944.

Reduce the samples to pass a No. 40 (840-micron) sieve.

(*b*) Weigh out, on the analytical balance, the following amounts of the samples: raw soil, 5 g.; soil-cement mixture, 5 g.; cement, 1 g. Place each of the weighed samples in a 250-ml. beaker. Add 50 ml. of HCl (1:1) (Note 1) to each sample, cover, and boil *gently* for 5 min. on the hot plate.

NOTE 1.—In the case of the cement sample, it is usually preferable first to add 40 ml. of water and then stir to obtain a thorough mixture. Then add 10 ml. of HCl (sp. gr. 1.18) and boil *gently* just long enough to obtain decomposition of the cement. Vigorous or extended boiling of soil or cement samples is seldom necessary, and often results in much slower filtration.

(*c*) Add 25 ml. of hot water to the beakers, stir, allow to settle momentarily, and then decant the contents through a Whatman No. 1 filter paper (Note 2), preferably 15 cm. in diameter. The filtrate should be received in a 250-ml. volumetric flask. When the liquid has passed through the filter paper, wash the residue once by decantation, using hot water; then transfer it to the filter, using a stream of hot water. The beaker should be rapidly policed, the loosened material being transferred to the filter paper. The material on the filter should then be washed an additional four times, each washing consisting of 10 to 15 ml. of hot water directed in a stream from the wash bottle. Very small amounts of residue will occasionally pass through the filter. These ordinarily may be disregarded.

NOTE 2.—In the case of the soil and soil-cement samples, the bulk of the residue sometimes slows filtration appreciably. No difficulty is usually encountered from cement samples, and, as a rule, soil samples may be filtered and washed in less than 30 min. Some soil-cement mixtures require more time, but, if this period exceeds 1 hr., subsequent filtration in similar cases may be more rapid if a No. 41 paper is substituted for the No. 1 paper. Slow filtration in such cases is generally caused by excessive boiling, resulting in gelation of the silica, which materially retards filtration.

(*d*) When washing has been completed, discard the filter, and dilute the filtrate in the volumetric flask to 250 ml. with cold water. The temperature of the solution should be near the calibration point of the flask. Agitate the flask to mix the contents thoroughly, then remove a 50-ml. aliquot and transfer to the original 250-ml. beaker (Paragraph (*b*)), using a 50-ml. pipette. Dilute to 100 ml. Make the solution slightly ammoniacal (Note 3), boil 1 to 2 min., and allow the hydroxides to settle.

NOTE 3.—If the samples contain ferrous iron it is desirable to add a few drops of HNO_3 before precipitation of the hydroxides.

(*e*) Filter the hydroxides through an 11-cm. Whatman No. 1 (or No. 41) filter paper, receiving the filtrate in the 600-ml. beaker. Wash the original 250-ml. beaker into the filter once with a stream of hot NH_4NO_3 (20 g. per l.), and follow by washing the hydroxide precipitate once or twice with hot NH_4NO_3 (20 g. per l.). Set the filtrate aside, and place the original beaker under the funnel. Perforate the paper with a rod (Note 4), and wash the hydroxides down into the original beaker, using a stream of hot NH_4NO_3 (20 g. per l.) to remove most of the precipitate from the filter paper. Treat the paper with 20 ml. of hot HCl (1:3), directing the acid over the paper with a glass rod. Wash the paper several times with hot water, and then discard the paper. Dilute the solution to 75 ml.

NOTE 4.—Instead of perforating the filter paper, the paper and precipitate may be transferred to the original beaker, the hydroxides dissolved with 20 ml. of hot HCl (1:3) and diluted to 75 ml. with water, and the procedure continued as described in Paragraph (*f*). In this case, the reprecipitated hydroxides and pulp are subsequently removed simultaneously.

(*f*) Make the solution slightly ammoniacal and boil 1 to 2 min. Allow the precipitate to settle, then decant through a Whatman No. 1 paper as before, receiving the filtrate in the 600-ml. beaker previously set aside (Paragraph (*e*)). Wash and police the beaker in which precipitation took place, finally washing the precipitate on the filter three or four times with NH_4NO_3 (20 g. per l.). Discard the hydroxide precipitate. Add 2 ml. of NH_4OH (sp. gr. 0.90) to the filtrate, which will now have a volume of 250 to 350 ml. Heat the solution to boiling and add 10 ml. of hot saturated ammonium oxalate solution. Keep the mixture near boiling until the precipitate becomes granular, then set aside on a warm hot plate for 30 min. or more. Before filtering off the calcium oxalate, verify completeness of precipitation, and make sure that a slight excess of NH_4OH is present. Filter the mixture through an 11-cm. or 15-cm. Whatman No. 2 filter paper, or if preferred a Whatman No. 42 paper, making sure that all the precipitate is being retained. Thoroughly clean with a rubber policeman the beaker in which precipitation took place, and transfer the contents to the filter with a stream of hot water. Wash the filter eight to ten times with hot water (not over 75 ml.) (Note 5) using a stream from the wash bottle.

NOTE 5.—The filter may be washed four times each with NH_4OH (2:98) and hot water, in the order stated.

(*g*) Transfer the filter paper and contents to a 400-ml. beaker containing 125 ml. of water and 6 ml. of H_2SO_4 (sp. gr. 1.84). If preferred, the precipitate may be removed from the paper by suitable means. Complete the determination in accordance with Paragraphs (*h*) and (*i*) or Paragraph (*j*).

(*h*) Heat the solution to 85 C. and titrate with standard 0.1 *N* $KMnO_4$.

(*i*) *Blank.*—Make a blank determination, following the same procedure and using the same amounts of all reagents.

(*j*) *Alternative Titration Procedure.*—Heat the solution to 85 C. and titrate as described in Paragraph (*h*), except that the $KMnO_4$ solution need not be a standard 0.1 *N* solution but the same solution shall be used in titrating all the components. Omit the blank determination described in Paragraph (*i*).

Calculations

6. Calculate the cement content of the soil-cement mixture as follows:

(*a*) When the determination has been completed in accordance with Section 5 (*h*) and (*i*):

(*1*) Calculate the percentages of CaO in the soil, the cement, and the soil-cement mixture as follows:

$$\text{CaO, per cent} = \frac{(A - B)C \times 0.0028}{D} \times 100$$

where:

A = milliliters of $KMnO_4$ solution required for titration of the sample,
B = milliliters of $KMnO_4$ solution required for titration of the blank,
C = normality of the $KMnO_4$ solution,
D = grams of sample represented by the aliquot titrated (Note 1), and
0.0028 = CaO equivalent of 1 ml. of 0.1 *N* $KMnO_4$.

(*2*) Calculate the percentage by weight of cement in the soil-cement mixture as follows (Note 2):

$$\text{Cement, per cent} = \frac{G - F}{E - F} \times 100$$

where:

E = percentage of CaO in cement,
F = percentage of CaO in raw soil, and
G = percentage of CaO in soil-cement mixture.

NOTE 1.—The aliquots titrated are equivalent to 1 g. of soil or soil-cement and 0.2 g. of cement.

NOTE 2.—When hydrated soil-cement mixtures are being analyzed, the value for percentage by weight of cement obtained in accordance with Section 6 (*a*) or (*b*) is in terms of hydrated cement. Such values may be converted to an approximate equivalent of dry cement by multiplying them by the factor 1.04.

(*b*) When the determination has been completed in accordance with Section 5 (*j*), calculate the percentage by weight of cement in the soil-cement mixture as follows (Note 2):

$$\text{Cement, per cent} = \frac{J - I}{5H - I} \times 100$$

where:

H = milliliters of $KMnO_4$ solution required for titration of the sample of cement,

I = milliliters of $KMnO_4$ solution required for titration of the sample of raw soil, and

J = milliliters of $KMnO_4$ solution required for titration of the sample of soil-cement mixture.

Tentative Definitions of

TERMS AND SYMBOLS RELATING TO SOIL MECHANICS[1]

A.S.T.M. Designation: D 653 – 42 T

ISSUED, 1942.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

PART I.—SYMBOLS AND TERMS RELATING TO SOIL MECHANICS.

SYMBOL	TERM
A	Area (II)[3]
a_v	Compressibility, coefficient of (V)
b	Breadth, or width (II)
C	Cohesion, resultant or total (IV, E)
C_c	Compression index (V)
C_s	Swelling index (V)
C_u	Uniformity coefficient (Hazen) (III)
c	Cohesion per unit area (IV, E)
c_v	Consolidation, coefficient of (V)
D	Diameter, or grain diameter, (II, III)
D_{10}	Effective size (Hazen) (III)
D_d	Density, degree of, or compaction, degree of (III)
E	Modulus of elasticity (IV, D)
e	Base of natural logarithms, or void ratio (II, III)
e_c	Void ratio, critical (III)
e_o	Void ratio at pressure p_o on the compression curve (V)
e_f	Flocculation ratio (III)
e_s	Void ratio at pressure p_o on the swelling curve (V)
F	Force, total internal (IV, A)
F_n	Normal component of F (IV, A)
F_t	Tangential (shearing) component of F (IV, A)
f	Force per unit area, or stress per unit area, internal (IV, A)
f_n	Normal component of f (IV, A)
f_t	Tangential (shearing) component of f (IV, A)
$\bar{F}$	Effective force, total internal (IV, A)
$\bar{F}_n$	Normal component of $\bar{F}$ (IV, A)
$\bar{F}_t$	Tangential (shearing) component of $\bar{F}$ (IV, A)
$\bar{f}$	Effective force per unit area, or effective stress per unit area, internal (IV, A)
$\bar{f}_n$	Normal component of $\bar{f}$ (IV, A)
$\bar{f}_t$	Tangential (shearing) component of $\bar{f}$ (IV, A)
G	Specific gravity (III)
g	Acceleration of gravity (II)
H	Depth, height, thickness (II)
H_s	Reduced height (II)
I	Modulus of incompressibility (IV, D)
I_c	Consistency index (III)
I_f	Flow index (III)
I_p	Plasticity index (III)
I_R	Remolding index (III)
I_T	Toughness index (III)
I_w	Moisture index (III)
i	Hydraulic gradient (II)

[1] Under the Standardization procedure of the Society, these definitions are under the jurisdiction of the A.S.T.M. Committee D-18 on Soils for Engineering Purposes.

[2] Accepted by Committee E-10 on Standards, May 20, 1942.

[3] The boldface roman numerals in parentheses indicate that a further definition of the symbol and term appears in a later part, that is, in Parts II to V, which cover separate classifications of the terms as to properties.

J Seepage force, resultant (II)
j Seepage force per unit volume (II)
K Hydrostatic pressure ratio (III)
k Permeability, coefficient of (III)
L Distance, or length (II)
L_E Lineal expansion (III)
L_{LE} Lineal expansion limit (III)
L_{LS} Lineal shrinkage limit (III)
L_S Lineal shrinkage (III)
m Perimeter shear (IV, E)
n Porosity, or developed pressure (II; III; IV, E)
P Force, total, or load, total (IV, B)
P_A Earth pressure, total active (IV, B)
P_P Earth pressure, total passive (IV, B)
p Force per unit area, or load per unit area (IV, B)
Q Discharge, rate of, or resistance, total or resultant (II; IV, E)
q Discharge, rate of per unit length or per unit area, or resistance per unit area or length (II; IV, E)
q_o Ultimate bearing capacity per unit area at ground surface (IV)
q_r Compressive strength per unit area (triaxial) (IV, E)
q_s Shear strength per unit area (IV, E)
q_u Compressive strength per unit area (unconfined test) (IV, E)
R Reading (II)
R_s Shrinkage ratio (III)
r Radius (II)
S Hydraulic gradient; or saturation, degree of (III)
s Shear strength per unit area (II; IV, E)
T Temperature (II)
T_v Time factor (V)
t Time (II)
U Neutral force (V)
$U\%$ Consolidation, percentage of (V)
u Hydrostatic excess pressure (V)
u_w Neutral pressure per unit area (V)
u_z Hydrostatic pressure (V)
V Volume (II)
V_S Volumetric shrinkage (III)
V_{SL} Volumetric shrinkage limit (II)
v Velocity (II)
W Weight, or load, total (II; IV, B)
$W\%$ Percentage finer matter in mechanical analysis plot (III)
w Water content (moisture content) (III)
w_{cme} Centrifuge moisture equivalent (III)
w_f Flocculation limit (III)
w_{fme} Field moisture equivalent (II)
w_l Liquid limit (III)
w_o Water content, optimum (III)
w_p Plastic limit (III)
w_s Shrinkage limit (III)
α Angle (II)
β Angle (II)
γ Unit weight (III)
γ_w Unit weight of water, equal to 1 in the metric system
δ Displacement, or deformation, total (see also ρ) (IV, C)
δ_x, δ_y, δ_z Components of δ (IV, C)
ϵ Base of natural logarithms, or deformation per unit length, or strain (II; IV, C)
ζ Component of ρ in z direction (IV, C)
η Component of ρ in y direction (II; IV, C)
θ Angle (II)
μ Coefficient of absolute viscosity
ν Coefficient of kinematic viscosity, or concentration factor (II; IV, A)
ξ Component of ρ in x direction (IV, C)
ρ Displacement, or deformation, total (see also δ) (IV, C)
ρ_x, ρ_y, ρ_z Components of ρ (IV, C)
σ Normal component of internal force per unit area (normal component of stress) (IV, A)
$\bar{\sigma}$ Normal component of effective internal force per unit area (normal component of stress) (IV, A)
τ Tangential component of internal force per unit area (tangential component of stress) (IV, A)
$\bar{\tau}$ Tangential component of effective internal force per unit area (tangential component of effective stress) (IV, A)
ϕ Angle of internal friction (III)
ψ Angle (II)

Greek Alphabet

Α α alpha	Η η eta	Ν ν nu	Τ τ tau
Β β beta	Θ θ ϑ theta	Ξ ξ xi	Υ υ upsilon
Γ γ gamma	Ι ι iota	Ο ο omicron	Φ φ ϕ phi
Δ δ delta	Κ κ kappa	Π π pi	Χ χ chi
Ε ε epsilon	Λ λ lambda	Ρ ρ rho	Ψ ψ psi
Ζ ζ zeta	Μ μ mu	Σ σ ς sigma	Ω ω omega

PART II.—GENERAL TERMS.

Acceleration of Gravity, g.

Angle, α, β, θ, ψ.—Angles are generally best designated by Greek letters. These four letters have no specific application in this list, and are therefore convenient for designating angles.

Area, A.

Base of Natural Logarithms, e or ϵ.—The symbol, e, is preferred, but where its use might cause confusion with its use designating void ratio the symbol, ϵ, should be used.

Breadth, b.

Coefficient of Absolute Viscosity, μ.

Coefficient of Dynamic Viscosity, ν.—Defined by the equation $\nu = \frac{\mu}{\gamma_f}$, where γ_f = the unit weight of the fluid in question.

Depth, H.

Diameter, D.

Discharge, Rate of, Q.—In soil mechanics literature this usually refers to the rate at which water is discharged through a given area of soil. Dimensionally, Q = volume/time. In Darcy's familiar equation for laminar flow $Q = kSA$.

Discharge, Rate of per Unit Length or per Unit Area, q.—Dimensionally, q = volume/(time × area), or q = volume/(time × length). In Darcy's equation as used above $q = kS$.

Distance, L.

Height, H.

Hydraulic Gradient, S or i.—The symbol, S, is preferred because of its widespread use in literature on hydraulics. The symbol, i, is included because it has been almost exclusively used to designate hydraulic gradient in soil mechanics literature.

Length, L.

Radius, r.

Reading, R.—For example, reading of a deflection dial, hydrometer, and similar apparatus.

Reduced Height, H_s.—The volume of the solid particles in a given soil mass divided by the cross-sectional area of the mass. Usually applied to a soil stratum where it represents the thickness or height which the stratum would have if its void ratio were zero. Expressed by the equation $H_s = \frac{H}{(1+e)}$. Note that in consolidation calculations the height and reduced height of a stratum draining at both faces are generally designated by $2H$ and $2H_s$, respectively.

Seepage Force, Resultant, J.

Seepage Force per Unit Volume, j.—Note that $j = S\gamma_f$, where γ_f is the unit weight of the fluid.

Temperature, T.

Thickness, H.

Time, t.

Velocity, v.

Volume, V.

Weight, W.

Width, b.

PART III.—TERMS USED IN THE DESCRIPTION OF SOILS AND THEIR PROPERTIES.

Angle of Internal Friction, ϕ.

Centrifuge Moisture Equivalent, w_{cme}.—In most uses the abbreviation *CME* may be used. When, as rarely, a symbol is desired, w_{cme} should be used. The *CME* is the water content retained by a soil

which has been first saturated with water and then subjected to a force equal to one thousand times the force of gravity for one hour.

Compaction, Degree of, D_d.—Also called degree of density. Defined by the equation:

$$D_d = \frac{\text{(Void ratio in loosest state)} - \text{(Void ratio of sample)}}{\text{(Void ratio in loosest state)} - \text{(Void ratio in densest state)}}$$

Consistency Index, I_c.—The ratio of the difference between the liquid limit and the natural water content to the difference between the liquid limit and the plastic limit. Defined by the equation:

$$I_c = \frac{w_L - w}{I_p}$$

Density, Degree of.—See Compaction, Degree of.

Diameter, Grain, D.—Also called grain size, or particle size. The size of grain, usually in millimeters, as determined by sieve analysis or wet mechanical analysis; hence not a true grain size except for spherical grains.

Effective Size (Hazen), D_{10}.—The grain size on a mechanical analysis curve corresponding to $W\,\% = 10$.

Equivalent Volumetric Change.—See Volumetric Shrinkage Limit.

Field Moisture Equivalent, w_{fme}.—In most uses the abbreviation *FME* may be used. When, as rarely, a symbol is desired w_{fme} should be used. The *FME* is the minimum water content at which a drop of water placed on a smoothed surface of a soil will not immediately be absorbed by the soil but will spread over the surface and give it a shiny appearance.

Flocculation Limit, w_f.—The water content of a soil when it is in the condition of a deflocculated sediment.

Flocculation Ratio, e_f.—The void ratio of a soil when it is in the condition of a deflocculated sediment.

Flow Index, I_f.—The slope of the flow curve. The flow curve is the locus of points obtained from a standard liquid limit test and plotted on a graph representing water content as ordinates on an arithmetic scale and number of blows as abscissas on a logarithmic scale. Thus the flow index is numerically equal to the difference between the water content at 10 and at 100 blows, or at 1 and at 10 blows.

Hydrostatic Pressure Ratio, K.—A term used in earth pressure computations to represent the ratio between the normal component of pressure at a point on a given plane and the pressure which would exist at the same point in a liquid of the same unit weight. Thus: $p_n = K\gamma_m H$.

Lineal Expansion, L_E.—The increase in one dimension, expressed as a percentage of that dimension at the shrinkage limit, of a soil mass when the water content is increased from the shrinkage limit to any given water content.

Lineal Expansion Limit, L_{LE}.—The increase in one dimension, expressed as a percentage of that dimension at the shrinkage limit, of a soil mass when the water content is increased from the shrinkage limit to the field moisture equivalent.

Lineal Shrinkage, L_S.—The decrease in one dimension, expressed as a percentage of that dimension originally, of a soil mass when the water content is reduced from the original percentage to the shrinkage limit.

Lineal Shrinkage Limit, L_{LS}.—The decrease in one dimension, expressed as a percentage of that dimension at the field moisture equivalent, of a soil mass when the water content is reduced from the field moisture equivalent to the shrinkage limit.

Liquid Limit, w_L.—In most cases the abbreviation *LL* may be used. When, as rarely, a symbol is desired, w_L should be used. The *LL* is the upper limit of the plastic state, expressed as the water content at which the flow curve (see Flow Index) intersects the "25 blows" ordinate.

Moisture Content.—See Water Content.

Moisture Index, I_w.—The ratio of the plasticity index to the difference between the natural water content and the plastic limit expressed by the equation:

$$I_w = \frac{I_p}{w - w_p}$$

Percentage Finer, $W\%$.—The ordinate of the mechanical analysis curve, representing, for a given soil, the percentage of particles by weight having diameters smaller than the value of D corresponding to $W\%$.

Permeability, Coefficient of, k.—The discharge velocity of flow of a fluid through a porous mass under a unit hydraulic gradient. Thus in Darcy's equation, $V = kSAt$, for laminar flow, $k = \frac{V}{At}$, where A is the gross area of the soil mass.

Plasticity Index, I_p.—In most cases the abbreviation PI may be used. When, as rarely, a symbol is desired I_p should be used. The PI is the numerical difference between the water content at the liquid limit and the water content at the plastic limit. $I_p = w_L - w_p$.

Plastic Limit, w_p.—In most cases the abbreviation PL may be used. When, as rarely, a symbol is desired, w_p should be used. The PL is the lower limit of the plastic state, expressed as the minimum water content at which a soil can be rolled into a thread $\frac{1}{8}$ in. in diameter without crumbling.

Porosity, n.—The ratio, expressed as a percentage, of the intergranular space in a given soil mass to the total volume of the soil mass. Porosity must be clearly differentiated from void ratio, e. The relation between the two terms is expressed by the equation: $n = \frac{e}{(1+e)}$.

Remolding Index, I_R.—The ratio of the modulus of elasticity (or deformation) of a soil in the undisturbed state to the modulus of elasticity (or deformation) of the soil in the remolded state.

Saturation, Degree of, S.—The ratio, expressed as a percentage, of the volume of water in a given soil mass to the total volume of intergranular space (voids). Thus: $S = \frac{V_w}{V_v} \times 100$.

Shrinkage Limit, w_s.—In most cases the abbreviation SL may be used. When, as rarely, a symbol is desired, w_s should be used. The SL is the maximum water content at which a reduction in water content will not cause a decrease in volume of the soil mass.

Shrinkage Ratio, R_s.—In most cases the abbreviation SR may be used. When, as rarely, a symbol is desired, R_s should be used. The SR is the ratio between a given volume change, expressed as a percentage of the dry volume, and the corresponding change in water content above the shrinkage limit, expressed as a percentage of the weight of the oven-dried soil.

Specific Gravity, G.—The ratio of the weight in air of a given volume of a material at a stated temperature to the weight in air of an equal volume of distilled water at a stated temperature, usually 4 C.

Toughness Index, I_T.—In most cases the abbreviation TI may be used. When, as rarely, a symbol is desired, I_T should be used. The TI is the ratio of the plasticity index to the flow index. Thus $I_T = \frac{I_p}{I_f}$.

Uniformity Coefficient (Hazen), C_u.—The ratio of D_{60} to D_{10} as determined in a mechanical analysis.

Unit Weight, γ.—Weight per unit volume, as grams per cubic centimeters, pounds per cubic foot, etc.

Void Ratio, e.—The ratio of the volume of intergranular space to the volume of solid particles in a given soil mass without regard to the proportions of liquid, air, or gas which may occupy the space.

Void Ratio, Critical, e_c.—The void ratio of a cohesionless soil at which continuous shearing deformation can take place without change in void ratio.

Volumetric Change.—See Volumetric Shrinkage.

Volumetric Shrinkage, V_S.—Also called volumetric change. The decrease in volume, expressed as a percentage of the dry volume, of a soil mass when the water content is reduced from a given percentage to the shrinkage limit.

Volumetric Shrinkage Limit, V_{SL}.—The decrease in volume, expressed as a percentage of the dry volume, of a soil mssa when the water content is reduced from the field moisture equivalent to the shrinkage limit.

Water Content, w.—The ratio, expressed as a percentage, of the weight of water in a given soil mass to the weight of solid particles.

Water Content, Optimum, w_o.—The water content at which the maximum density is produced in a soil by a specific amount of compaction.

PART IV.—TERMS RELATING TO STRESS, STRAIN, AND DISPLACEMENT.

Section A: Internal Stresses and Forces

Concentration Factor, ν.—Factor used in Frölich's and Griffith's theory of stress distribution, as in the equation:

$$f_z = \frac{\nu P}{2\pi R^2}\left(\frac{z}{R}\right)^{\nu}$$

Force, Total Internal, F.—The total force acting on a given area within a body or within a mass of earth.

Force per Unit Area, or Stress per Unit Area, Internal, f.—The force per unit area or stress per unit area acting at a point or on a surface within a body or within a mass of earth.

Effective Force, Total Internal, $\bar{F}$.—The total force acting on the soil structure over a given area within a body or within a mass of earth, for example, $\bar{F} = F - U$.

Effective Force per Unit Area, or Effective Stress per Unit Area, Internal, $\bar{f}$.—The force per unit area or stress per unit area acting on the soil structure at a point or on a surface within a body or within a mass of earth, for example, $\bar{f} = f - u$.

Normal Component of These Forces and Stresses, F_n, $\bar{F}_n$, f_n, or σ; $\bar{f}_n$ or $\bar{\sigma}$.

Tangential (or Shearing) Component of These Forces and Stresses, F_t, $\bar{F}_t$, f_t or τ; $\bar{f}_t$ or $\bar{\tau}$.

Section B: External Forces, Pressures, and Loads

Earth Pressure, Total Active, P_A.—The total force exerted by or through a mass of earth against a medium of lateral support or against a given area thereof.

Earth Pressure, Total Passive, P_P.—The total externally applied lateral force which a given mass of earth is capable of resisting over a given area.

Force, Total, P.—Externally applied force.

Force per Unit Area, p.—Intensity of externally applied force.

Load, Total, P or W.—Externally applied load.

Load per Unit Area, p.—Intensity of externally applied load.

Section C: Strains and Displacements

Displacement, δ or ρ.—Translation of a body or mass through space.

Deformation, Total, δ or ρ.—Total deformation of a body or mass within itself, usually caused by externally applied forces.

Components of Displacement or Total Deformation.—The following are used to to identify components of displacement or total deformation:

Components in the x-Direction, δ_x or ρ_x or ξ.

Components in the y-Direction, δ_y or ρ_y or η.

Components in the z-Direction, δ_z or ρ_z or ζ.

Deformation per Unit Length, ϵ.

Strain, ϵ.

Section D: Moduli

Modulus of Elasticity, E.—The ratio of stress to strain for a given material under given load conditions. Subscripts may be used to differentiate moduli under different load conditions.

Modulus of Incompressibility, I.—The ratio of the developed pressure to the volume change in a soil mass of unit volume.

Section E.: Resistances and Strengths

Cohesion per Unit Area, c.—The shearing strength per unit area under zero externally applied load, as used in Rankine's equation: $s = c + p \tan \phi$. The value of c is not a constant for a given soil, but varies according to the test procedure.

Cohesion, Resultant or Total, C.—The total cohesion on a given area, that is, $C = cA$.

Developed Pressure, n.—The stress reaction of a soil mass acting as a pressure per unit area independent of the dimension of the loaded area, and consisting of pressure, components developed by the mass due to resistance to displacement.

Perimeter Shear, m.—The boundary stress reaction per unit length of perimeter developed by a loaded area, a measure of that proportion of the applied pressure which is carried by the body of soil as a function of the perimeter of the bearing area.

Resistance, Total Resultant, Q.—A general symbol to be used with appropriate subscripts where necessary.

Resistance per Unit Area or Length, q.—A general symbol to be used with appropriate subscripts where necessary.

Compressive Strength per Unit Area, q_u.—Compressive strength as determined by an unconfined compression test.

Compressive Strength per Unit Area, q_r.—Compressive strength as determined by a triaxial compression test. Note that $q_r = \sigma_I - \sigma_{III}$ at failure.

Shear Strength per Unit Area, q_s or s.

Ultimate Bearing Capacity per Unit Area at Ground Surface, q_o.—In an homogeneous soil, the ultimate bearing capacity increases with depth. Ultimate bearing capacities at various depths may be designated by q with suitable subscripts.

PART V.—TERMS RELATING TO THE CONSOLIDATION OF SOILS.

NOTE.—Readers of literature published prior to 1935 should observe that the coefficient of consolidation, usually designated by c, was then defined by the equation $c = \frac{k}{a_v \gamma_w (1 + e)}$. This original definition of coefficient of consolidation may be found in some more recent papers and care should be taken to avoid confusion.

Compressibility, Coefficient of, a_v.—Numerically equal to the slope of the pressure-void ratio diagram obtained from a consolidation test, expressed by the equation: $a_v = -\frac{de}{dp}$.

Compression Index, C_c.—Compression index for a clay sample in the undisturbed state expressed by the equation: $e = e_o - C_c \log \frac{p}{p_o}$. Use C_c' for a clay sample in the remolded state.

Swelling Index, C_s.—Swelling index for a clay sample in the undisturbed state, expressed by the equation: $e = e_s - C_s \log \frac{p}{p_s}$. Use C_s' for a clay sample in the remolded state.

Void ratio at pressure p_o (usually 1 kg. per sq.cm.) on the compression curve obtained by a consolidation test on a clay sample in the undisturbed state, e_o. Use e_o' for a clay sample in the remolded state.

Void ratio at pressure p_s (usually 1 kg. per sq.cm.) on the swelling curve obtained by a consolidation test on a clay sample in the undisturbed state, e_s. Use e_s' for a clay sample in the remolded state.

Consolidation, Coefficient of, c_v.—Defined by Terzaghi by the equation:

$$c_v = \frac{k(1 + e)}{a_v \gamma_w}$$

Consolidation, Percentage of, $U\%$.—Average degree of theoretical consolidation at a given time within a clay stratum, expressed as a percentage of the total consolidation obtainable under the given stress condition.

Time Factor, T_v.—A dimensionless factor used in the calculation of the rate of consolidation. The value of T_v corresponding to a given percentage consolidation, $U\%$, depends solely on the distribution of stress increment within the consolidating stratum.

Hydrostatic Pressure, u_z.

Neutral Pressure per Unit Area, u_w.—The total pressure per unit area of soil in the pore water at a given time.

Neutral Force, U.—The total pressure, over a given area of soil, in the pore water at a given time expressed by the equation: $U = u_w A$.

Hydrostatic Excess Pressure, u.—The pressure per unit area of soil which exists in the pore water at any time in excess of the hydrostatic pressure, expressed by equation: $u = u_w - u_z$. Note also that $u = f - \bar{f}$.

Tentative Specifications for

ASPHALT FOR DAMPPROOFING AND WATERPROOFING[1]

A.S.T.M. Designation: D 449 – 42 T

ISSUED, 1937; REVISED, 1942.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These specifications cover three types of asphalt suitable for use as a mopping coat in dampproofing, or as a plying or mopping cement in the construction of a membrane system of waterproofing.

Primer

2. The material used as a primer shall conform to the Standard Specifications for Primer for Use with Asphalt in Dampproofing and Waterproofing (A.S.T.M. Designation: D 41) of the American Society for Testing Materials.[3]

Membrane Materials

3. For the construction of a membrane system of waterproofing, any or all of the following felts or fabrics conforming to the specifications of the American Society for Testing Materials may be used alone or in various combinations:

(*a*) *Felt.*—Standard Specifications for Asphalt-Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs (A.S.T.M. Designation: D 226)[3]

(*b*) *Asbestos Felt.*—Tentative Specifications for Asphalt-Saturated Asbestos Felts for Use in Waterproofing and in Constructing Built-Up Roofs (A.S.T.M. Designation: D 250).[3]

(*c*) *Cotton Fabrics.*—Standard Specifications for Woven Cotton Fabrics Saturated with Bituminous Substances for Use in Waterproofing (A.S.T.M. Designation: D 173),[3] asphalt type.

Types

4. The asphalts covered by these specifications are of three types, as follows:

Type A.[4]—A somewhat less susceptible asphalt with good adhesive and "self-healing" properties for use above ground level where not exposed to temperatures exceeding 125 F. (52 C.).

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] These specifications comprise a revision and consolidation of and replace the former Tentative Specifications for Asphalt for Use in Dampproofing and Waterproofing Below Ground Level (D 40 – 36 T), and Tentative Specifications for Asphalt for Use in Dampproofing and Waterproofing Above Ground Level (D 144 – 36 T), which were accordingly discontinued in 1937.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

[4] Attention is called to the fact that when these specifications were editorially revised and rearranged in 1943 the designations of grades A and B were interchanged. The present grade A was formerly designated grade B, and grade B was designated grade A in the standard as published in 1942 Book of A.S.T.M. Standards, Part II, p. 1290.

NOTE.—This type of asphalt is suitable for railroad bridges, culverts, retaining walls, tanks, dams, conduits, spray decks, etc.

Type B.[4]—A soft, adhesive "self-healing" asphalt which flows easily under the mop and which is suitable for use below ground level under uniformly moderate temperature conditions both during the process of installation and during service.

NOTE.—This type of asphalt is suitable for foundations, tunnels, subways, etc.

Type C.—An asphalt less susceptible to temperature than type *A*, with good adhesive properties for use above ground level where exposed on vertical surfaces in direct sunlight or at temperatures above 125 F. (52 C.).

TABLE I.—REQUIREMENTS FOR ASPHALT FOR DAMPPROOFING AND WATERPROOFING.[4]

	Type *A*[4]		Type *B*[4]		Type *C*	
	Min.	Max.	Min.	Max.	Min.	Max.
Softening point (ring-and-ball method)	145 F. (63 C.)	170 F. (77 C.)	115 F. (46 C.)	145 F. (63 C.)	170 F. (77 C.)	200 F. (93 C.)
Flash point (Cleveland open cup)	400 F. (205 C.)	....	350 F. (175 C.)	....	400 F. (205 C.)	....
Penetration:						
0 C. (32 F.), 200 g., 60 sec.	10	....	5	....	10	....
25 C. (77 F.), 100 g., 5 sec.	25	50	50	100	20	40
46 C. (115 F.), 50 g., 5 sec.	....	115	100	....	....	100
Ductility at 25 C. (77 F.), (5 cm. per min.), cm.	15	....	30	....	2.5	....
Loss on heating at 163 C. (325 F.), 50 g., 5 hr., per cent.	....	1	....	2	....	1
Penetration of residue, per cent of original	60	....	60	....	60	....
Total bitumen soluble in carbon disulfide, per cent:						
Filled or native asphalt	....	....	95	....	....	....
Unfilled asphalt	99	....	99	....	99	....
Proportion of bitumen soluble in carbon tetrachloride, per cent	99	....	99	....	99	....
Ash, per cent:						
Filled or native asphalt	....	....	....	5	....	....
Unfilled asphalt	....	1	....	1	....	1
Coarse particles retained on No. 200 (74-micron) sieve as percentage of bitumen insoluble in carbon disulfide, per cent	....	....	....	12	....	....

Properties

5. The asphalt shall be homogeneous and free from water, and shall conform to the requirements prescribed in Table I.

Methods of Sampling and Testing

6. The asphalt shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(*a*) *Sampling.*—Tentative Methods of Sampling Bituminous Materials (A.S.T.M. Designation: D 140).[3]

(*b*) *Softening Point.*—Standard Method of Test for Softening Point of Bituminous Materials (Ring-and-Ball Method) (A.S.T.M. Designation: D 36).[3]

(*c*) *Flash Point.*—Standard Method of Test for Flash and Fire Points by Means of Open Cup (A.S.T.M. Designation: D 92).[3]

(*d*) *Penetration.*—Standard Method of Test for Penetration of Bituminous Materials (A.S.T.M. Designation: D 5).[3]

(*e*) *Ductility.*—Standard Method of Test for Ductility of Bituminous Materials (A.S.T.M. Designation: D 113).[3]

(*f*) *Loss on Heating.*—Tentative Method of Test for Loss on Heating of Oil and Asphaltic Compounds (A.S.T.M. Designation: D 6).[3]

(*g*) *Bitumen Soluble in Carbon Disulfide.*—Standard Method of Test for Determination of Bitumen (A.S.T.M. Designation: D 4).[3]

(*h*) *Bitumen Soluble in Carbon Tetrachloride.*—Standard Method of Test for Proportion of Bitumen Soluble in Carbon Tetrachloride (A.S.T.M. Designation: D 165).[3]

(*i*) *Ash.*—Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (A.S.T.M. Designation: D 271).[5]

(*j*) *Coarse Particles.*—Standard Method of Test for Coarse Particles in Mixtures of Asphalt and Mineral Matter (A.S.T.M. Designation: D 313).[3]

[5] 1944 Book of A.S.T.M. Standards, Part III.

Tentative Specifications for

COAL-TAR PITCH FOR STEEP BUILT-UP ROOFS[1]

A.S.T.M. Designation: D 654 – 42 T

ISSUED, 1942.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These specifications cover coal-tar pitch suitable for use as a mopping coat and for embedding slag or gravel in the construction of steep built-up roofs with inclines of 1 to 6 in. per horizontal foot, where nailing is employed. This pitch is also suitable for embedding and coating the top ply of felt on flat built-up promenade roofs, either level or with inclines up to 2 in. per horizontal foot, that are to be surfaced with quarry tile or other rigid or semirigid wearing surfaces.

NOTE.—Other types of coal-tar pitch for flat built-up roofs, dampproofing, and waterproofing are covered by the Standard Specifications for Coal-Tar Pitch for Roofing, Dampproofing, and Waterproofing (A.S.T.M. Designation: D 450) of the American Society for Testing Materials.[3]

Types

2. The coal-tar pitch covered by these specifications is of one type.

Primer

3. Steep pitch usually can be applied directly to concrete surfaces without priming, but, where priming is required, the material used as a primer shall be creosote conforming to the Standard Specifications for Creosote for Priming Coat with Coal-Tar Pitch in Dampproofing and Waterproofing (A.S.T.M. Designation: D 43) of the American Society for Testing Materials.[3]

Membrane Materials

4. For the construction of steep built-up roofs, felts conforming to the Standard Specifications for Coal-Tar Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs (A.S.T.M. Designation: D 227) of the American Society for Testing Materials[3] shall be used.

Properties

5. The coal-tar pitch shall be homogenous and shall conform to the requirements prescribed in Table I.

Sampling and Testing

6. The coal-tar pitch shall be sampled and the properties enumerated in these

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] Accepted by the Society at annual meeting, June, 1942.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

TABLE I.—REQUIREMENTS FOR COAL-TAR PITCH FOR STEEP ROOFS.

	Min.	Max.
Specific gravity, 25/25 C. (77/77 F.)	1.20	1.35
Softening point (ring-and-ball method)	150 F. (65 C.)	170 F. (77 C.)
Total bitumen soluble in carbon disulfide, per cent	65	85
Ash, per cent	...	2.0
Distillation Test:		
Total distillate by weight, 32 to 572 F. (0 to 300 C.), per cent	...	8
Residue by weight, per cent	92	...
Flash point (Cleveland open cup)	275 F. (135 C.)	...
Penetration:		
32 F. (0 C.), 200 g., 60 sec.	4	...
77 F. (25 C.), 100 g., 5 sec.	12	35
115 F. (46 C.), 50 g., 5 sec.	...	100

specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(*a*) *Sampling.*—Tentative Methods of Sampling Bituminous Materials (A.S.T.M. Designation: D 140).[3]

(*b*) *Specific Gravity.*—Standard Method of Test for Specific Gravity of Asphalts and Tar Pitches Sufficiently Solid to be Handled in Fragments (A.S.T.M. Designation: D 71).[3]

(*c*) *Softening Point.*—Standard Method of Test for Softening Point of Bituminous Materials (Ring-and-Ball Method) (A.S.T.M. Designation: D 36).[3]

(*d*) *Bitumen Soluble in Carbon Disulfide.*—Standard Method of Test for Determination of Bitumen (A.S.T.M. Designation: D 4).[3]

(*e*) *Ash.*—Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (A.S.T.M. Designation: D 271.).[4]

(*f*) *Distillation.*—Standard Method of Test for Distillation of Tar Products Suitable for Road Treatment (A.S.T.M. Designation: D 20).[3]

(*g*) *Flash Point.*—Standard Method of Test for Flash and Fire Points by Means of Open Cup (A.S.T.M. Designation: D 92).[3]

(*h*) *Penetration.*—Standard Method of Test for Penetration of Bituminous Materials (A.S.T.M. Designation: D 5).[3]

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[4] 1944 Book of A.S.T.M. Standards, Part III.

Tentative Specifications for

ASPHALT-SATURATED ASBESTOS FELTS FOR USE IN WATERPROOFING AND IN CONSTRUCTING BUILT-UP ROOFS[1]

A.S.T.M. Designation: D 250 - 44 T

ISSUED, 1942; REVISED, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These specifications cover asphalt-saturated asbestos felts, either 36 or 32 in. in width, composed of asbestos felt saturated, but not coated, with asphalt for use in the membrane system of waterproofing and in the construction of built-up roofs.

Types

2. Asphalt-saturated asbestos felts covered by these specifications are of two types: namely,

15-lb. Type
30-lb. Type

Mopping Materials

3. For the construction of built-up roofs, asphalt mopping materials conforming to the Standard Specifications for Asphalt for Use in Constructing Built-Up Roof Coverings (A.S.T.M. Designation: D 312)[3] shall be used.

Manufacture

4. In the process of manufacture, a single thickness of asbestos felt shall be saturated with an asphaltic saturant.

Character of Felt

5. The felt shall be an asbestos felt produced by "felting" at least 85 per cent by weight of asbestos fiber. The surface of the felt shall be uniformly smooth. Upon splitting or tearing on the bias, the felt shall appear reasonably free from lumps or particles of foreign substances.

Character of Saturant

6. The bitumen of the saturant shall be composed of asphaltic materials.

Character of Saturated Felt

7. The felt shall be thoroughly and uniformly saturated, and shall show no unsaturated spots at any point upon cutting 2-in. strips at random across the

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] Prior to their present publication as tentative, these specifications were published as tentative from 1926 to 1927, being revised in 1927. They were adopted in 1927, published as standard from 1927 to 1942, but revised and reissued as tentative in 1942.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

entire sheet and splitting them open for their full length.

Surface Finish

8. The surface of the felt shall not be coated or covered with talc or other substance that would tend to interfere with the adhesion between the felt and the plying cement.

Physical Properties

9. The fabricated product shall conform to the following requirements:

	15-lb. Type	30-lb. Type
Width of roll	either 36 or 32 in., as may be specified, ± ½ in.	
Area of roll, min. sq. ft.	324	216
Minimum weight of saturated felt per 100 sq. ft. (exclusive of wrapping and packing material), lb	13	30
Loss on heating at 221 F. (105 C.) for 5 hr. max., per cent	5	5
Pliability at 77 F. (25 C.): At least 8 out of 10 strips shall not crack when bent 90 deg. at a uniform speed over a rounded corner of	½ in. rad.	¾ in. rad.
Saturant in moisture-free felt, min., per cent by weight	40	50

Desaturated Felt

10. The desaturated moisture-free felt shall conform to the following requirements:

	15-lb. Type	30-lb. Type
Weight per 100 sq. ft., min., lb	9.0	17.5
Ash, min., per cent	70.0	70.0
Thickness, min., in.	0.020	0.040

Freedom from Defects

11. The finished material shall be free of visible external defects, such as holes, ragged or untrue edges, breaks, cracks, tears, protuberances, and indentations.

Pliability and Stickiness

12. The rolls shall not crack nor be so sticky as to cause tearing or material damage upon being unrolled at atmospheric temperatures above 50 F. (10 C.).

Packing

13. The rolls of saturated felt need not be wound on cores, but they shall be securely wrapped in a substantial grade of paper of the same width as the fabric. The wrapper shall completely encircle the roll and shall be pasted at the overlap in a manner that will prevent it shifting from position. The ends of the roll need not be covered. As an alternative, the rolls of saturated felt may either be tied with strings or simply pasted at the ends, as may be agreed upon by the purchaser and the seller. No roll shall contain more than two pieces, and there shall not be more than 3 per cent of rolls containing two pieces in any shipment.

Marking

14. Each roll shall be plainly marked with the manufacturer's name and brand or as agreed upon by the seller and the purchaser, and shall bear a notation showing the type of saturant.

Sampling and Testing

15. The material shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the Tentative Methods of Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing and Roofing (A.S.T.M. Designation: D 146) of the American Society for Testing Materials.[3]

Inspection

16. Inspection of material shall be made as agreed upon by the seller and the purchaser as part of the purchase contract.

Basis of Rejection

17. Failure to conform to any one of the requirements prescribed in these specifications shall constitute grounds for rejection. In case of rejection, the seller shall have the right to reinspect the rejected shipment and resubmit the lot after removal of those packages not conforming to the specified requirements.

Tentative Specifications for

ASPHALT-SATURATED AND COATED ASBESTOS FELTS FOR USE IN CONSTRUCTING BUILT-UP ROOFS[1]

A.S.T.M. Designation: D 655 – 44 T

ISSUED, 1942; REVISED, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These specifications cover asphalt-saturated and asphalt-coated asbestos felts in sheet form for use in the construction of built-up roofs. They may be either 36 or 32 in. in width and shall be composed of asbestos roofing felt saturated and coated on one or both sides with asphalt. The coated sides shall be surfaced with mineral matter.

Types

2. Asphalt-saturated and asphalt-coated asbestos felts covered by these specifications are of two types: namely,

20-lb. Type
50-lb. Type

Mopping Materials

3. For the construction of built-up roofs, asphalt mopping materials conforming to the Standard Specifications for Asphalt for Use in Constructing Built-Up Roof Coverings (A.S.T.M. Designation: D 312)[3] shall be used.

Manufacture

4. In the process of manufacture, a single thickness of asbestos felt shall be saturated with an asphaltic saturant, coated on one side for the 20-lb. type and on both sides for the 50-lb. type with asphalt, and surfaced with sufficient mineral matter to prevent sticking in the rolls.

Character of Felt

5. The felt shall be an asbestos felt produced by "felting" at least 85 per cent by weight of asbestos fiber. The surface of the felt shall be uniformly smooth. Upon splitting or tearing on the bias, the felt shall appear reasonably free from lumps or particles of foreign substances.

Character of Saturant and Coatings

6. The bitumen of the saturant and coatings shall be composed of asphaltic materials.

Character of Saturated Felt

7. The felt shall be thoroughly and uniformly saturated and shall show no

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] Revision accepted by the Society at annual meeting, June, 1944.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

unsaturated spots at any point upon cutting 2-in. strips at random across the entire sheet and splitting them open for their full length.

Surface Finish

8. The surface of the felt shall be coated on one side for the 20-lb. type and on both sides for the 50-lb. type with asphalt, and surfaced with fine sand or other mineral matter that will prevent sticking in the rolls but will not interfere with the adhesion between the felt and the plying cement.

Physical Properties

9. The fabricated product shall conform to the following requirements:

	20-lb. Type	50-lb. Type
Width of roll	either 36 or 32 in., as may be specified, ± ½ in.	
Area of roll, min., sq. ft.	108	108
Minimum weight of finished product per 100 sq. ft. (exclusive of wrapping and packing material), lb	17	50
Loss on heating at 221 F. (105 C.) for 5 hr., max., per cent	5	5
Pliability at 77 F. (25 C.): At least 8 out of 10 strips shall not crack when bent 90 deg. at a uniform speed over a rounded corner of	½ in. rad.	1 in. rad.
Saturant in moisture-free felt, min., per cent by weight	40	50
Weight of mineral surfacing per 100 sq. ft., max., lb	3	10

Desaturated Felt

10. The desaturated moisture-free felt shall conform to the following requirements:

	20-lb. Type	50-lb. Type
Weight per 100 sq. ft., min., lb	8.5	17.5
Ash, min., per cent	70.0	70.0
Thickness, min., in.	0.020	0.040

Freedom from Defects

11. The finished material shall be free of visible external defects, such as holes, ragged or untrue edges, breaks, cracks, tears, protuberances, and indentations.

Pliability and Stickiness

12. The rolls shall not crack nor be so sticky as to cause tearing or material damage upon being unrolled at atmospheric temperatures above 50 F. (10 C.).

Packing

13. The rolls of saturated and coated felt need not be wound on cores, but they shall be securely wrapped in a substantial grade of paper of the same width as the fabric. The wrapper shall completely encircle the roll and shall be pasted at the overlap in a manner that will prevent it shifting from position. The ends of the roll need not be covered. As an alternative, the rolls may either be tied with strings or simply pasted at the ends, as may be agreed upon by the seller and the purchaser. No roll shall contain more than two pieces, and there shall not be more than 3 per cent of rolls containing two pieces in any shipment.

Marking

14. Each roll shall be plainly marked with the manufacturer's name and brand or as agreed upon by the seller and the purchaser, and shall bear a notation showing the type of saturant.

Sampling and Testing

15. The material shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the Tentative Methods of Testing Asphalt Roll Roofing, Cap Sheets, and Shingles (A.S.T.M. Designation: D 228) of the American Society for Testing Materials,[3] with the following exceptions:

(*1*) The weight of material exclusive of packing and wrapping and the weight of mineral surfacing shall be determined on the basis of 100 sq. ft. instead of 108 sq. ft.

(*2*) The loss on heating shall be determined at 221 F. (105 C.) instead of 176 F. (80 C.).

Inspection

16. Inspection of material shall be made as agreed upon by the purchaser and the seller as part of the purchase contract.

Basis of Rejection

17. Failure to conform to any one of the requirements prescribed in these specifications shall constitute grounds for rejection. In case of rejection, the seller shall have the right to reinspect the rejected shipment and resubmit the lot after removal of those packages not conforming to the specified requirements.

Tentative Specifications for
ASPHALT ROOFING SURFACED WITH COARSE MINERAL GRANULES[1]

A.S.T.M. Designation: D 249 - 44 T

ISSUED, 1933; REVISED, 1934, 1935, 1937, 1941, 1942, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These specifications cover asphalt roofing in sheet form surfaced with colored mineral granules, either 36 or 32 in. in width, composed of roofing-felt saturated and coated on both sides with asphalt and surfaced on the weather side with granulated slate or equivalent mineral material (of solid or mixed colors as may be agreed upon by the purchaser and the seller), and on the reverse side with a suitable material to prevent sticking in the package.

Manufacture

2. In the process of manufacture, a single thickness of dry roofing felt shall be impregnated with a hot asphaltic saturant, then coated on both sides with a hot asphaltic coating compounded with a fine mineral filler substantially insoluble in water, and finally surfaced on the weather side with mineral granules embedded in the hot asphaltic coating. The reverse side shall be covered with suitable material to prevent the roofing from sticking in the package.

Character of Felt

3. The felt shall be a roofing felt produced by "felting" vegetable or animal fibers or mixtures thereof. The surface of the felt shall be uniformly smooth. Upon splitting or tearing on the bias, the felt shall appear reasonably free from lumps of underbeaten stock (that is, stock that has not been beaten or shredded into fiber in the process of manufacture) and particles of foreign substances (that is, fragments of stone, metal, leather, rubber, etc.).

Character of Saturant and Coatings

4. The bitumen of the saturant and coatings shall be composed of asphaltic materials.

Character of Saturated Felt

5. The felt shall be completely and uniformly saturated, and shall show no

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] Latest revision accepted by Committee E-10 on Standards, August 28, 1944.

Prior to their present publication as tentative, these specifications were published as tentative from 1925 to 1927. They were adopted in 1927, published as standard from 1927 to 1933, but withdrawn and republished as tentative in 1933.

Emergency Alternate Provisions EA - D 249b applying to this standard appear in the pink section of this book.

unsaturated spots at any point upon cutting 2-in. strips at random across the entire sheet and splitting them open for their full length.

Surface Finish

6. (*a*) The surface of the weather side shall be uniform in finish and texture. The mineral granules shall be uniformly distributed over the entire surface (except as noted in Paragraph (*b*)) in a smooth layer and shall be firmly embedded in the asphalt coating.

Physical Properties

7. The fabricated product shall conform to the requirements prescribed in Table I.

Freedom from Defects

8. The finished material shall be free of visible external defects, such as holes, ragged or untrue edges, rents, cracks, indentations, and lumps of coating.

TABLE I.—PHYSICAL REQUIREMENTS OF ASPHALT ROOFING.

	Max., lb.	Min., lb.
Width		The roofing shall be put up in rolls and shall be either 36 or 32 in. in width, as may be specified, within a permissible variation of ±1/4 in.
Area		The average area of the rolls examined shall contain sufficient roofing to cover 100 sq. ft. of roof surface (108 sq. ft. with 2 in. or no bare lapping edge, or 111 sq. ft. with 3 in. lapping edge).
Pliability at 77 F. (25 C.)		At least 8 strips out of 10 shall not crack when bent 90 deg. at a uniform speed over a rounded corner of 3/4-in. radius.
Behavior on heating to 176 F. (80 C.) for 2 hr		Not more than 1.5 per cent volatile matter loss. The granular surfacing shall not slide more than 1/16 in. when suspended vertically.
Weight per roll, net average:		
No bare edge	....	83.0
2 or 3-in. bare edge	....	81.0
Weight of dry felt per 108 sq. ft.	....	10.8
Weight of saturant (soluble in CS_2) per 108 sq. ft.[a]	....	17.3
Weight of weather side mineral filled coating per 108 sq. ft	35.0	16.0
Weight of mineral matter per 108 sq. ft. passing No. 6 (3360-micron) and retained on No. 100 (149-micron) sieve	....	20.0
Percentage by weight of mineral matter passing No. 100 (149-micron) sieve on the basis of the sum of the bitumen in the coatings and the mineral matter passing No. 100 (149-micron) sieve	50.0 per cent	15.0 per cent
Weight of any 108 sq. ft. roll of roofing in the shipment (coarse granule surfaced portion)	....	80.0

[a] The weight of saturant per 108 sq. ft. shall be not less than 1.6 times the weight of the dry felt.

(*b*) The coating and granules shall cover the entire weather side of the roofing, except that this side may be provided with a bare lapping edge, approximately 2 or 3 in. in width along one edge, where either the granules alone or both the granules and coating may be omitted. When the granules alone are omitted, a thin fibrous membrane may be used to prevent the coated lapping edge from sticking in the roll.

(*c*) The reverse side of the sheet shall have the asphalt coating and powdered surfacing applied uniformly to the edges.

Pliability and Stickiness

9. The finished product shall not crack nor be so sticky as to cause tearing or material damage upon being unrolled at atmospheric temperatures above 50 F. (10 C.)

Packing

10. (*a*) The roofing shall be put up in rolls of either 108 sq. ft. when made with a 2-in. or no bare-lapping edge, or 111 sq. ft. when made with a 3-in. bare lapping edge. No roll shall contain more than two pieces nor shall there be more

than 3 per cent of rolls containing two pieces in any shipment. The rolls shall be tightly wound, and shall be securely wrapped with a substantial grade of paper completely encircling the roll and pasted at the overlap in such a manner as to prevent shifting from position. The nails and lap-cement shall be packed in the core of each roll or furnished separately if so specified by the purchaser.

(*b*) Special packing shall be as agreed upon by the purchaser and the seller.

Marking

11. Each roll shall be plainly marked with the name and brand of the manufacturer.

Nails and Lap-Cement

12. (*a*) *Nails:* (*1*) *Dimensions.*—The gage of the wire shall be not greater than Washburn & Moen No. 10 (0.135 in.) nor less than Washburn & Moen No. 12 (0.105 in.). The head shall be not less than $\frac{3}{8}$ in. in diameter nor less than 0.025 in. in thickness. The shank shall be not less than $\frac{3}{4}$ in. in length and shall be smooth, barbed, or square and twisted (spiral). If barbed, the barbing shall not increase the diameter of the lower two thirds more than 0.01 in. The lower end of the shank shall be pointed. If square and twisted (spiral), the cross-sectional area shall be equivalent to that specified above.

(*2*) *Protective Coating.*—Nails shall be galvanized or sherardized, and shall resist four 1-min. dips in a copper sulfate solution when tested in accordance with the Standard Method of Test for Uniformity of Coating by the Preece Test (Copper Sulfate Dip) on Zinc-Coated (Galvanized) Iron or Steel Articles (A.S.T.M. Designation: A 239) of the American Society for Testing Materials.[3]

(*3*) *Quantity.*—There shall be furnished per "square" an average of not less than 252 nails for 36-in. roofing and not less than 275 nails for 32-in. roofing. Where the roofing is intended for application by the blind nailing method and is provided with a 3-in. selvage, 150 nails per "square" shall be furnished on a basis of 36-in. roofing.

(*b*) *Lap-Cement:* (*1*) *Lap-Cement for Exposed Nail Application.*—The cement shall be composed of bituminous materials dissolved in a volatile solvent, and shall be of such a nature as to firmly bind the laps without injurious effect on the roofing. The cement shall have a minimum flash point of 71 F. (21.5 C.) when tested by the Tag closed-cup tester.[4] There shall be furnished for each "square" of 36-in. roofing for exposed nail application not less than $\frac{3}{4}$ pt. of cement and for 32-in. roofing not less than 1 pt. of cement. The addition of 5 to 20 per cent by weight of short-fiber asbestos shall be permitted, in which case the quantity of cement shall be increased by $\frac{1}{4}$ pt. per "square."

(*2*) *Lap Cement for Concealed Nail Application.*—The cement shall be composed principally of bituminous materials dissolved in a volatile solvent. The cement shall have a minimum flash point of 71 F. (21.5 C.) when tested by the Tag closed-cup tester.[4] A strength-of-lap test shall be made, using roofing and cement from the same manufacturer or from the same shipment. The tensile strength of the cemented laps shall conform to the requirements prescribed in Table II. The test specimen shall consist of two strips each 6 in. in length and 1 in. in width cut from the roofing, and one of the strips shall include a 3-in. bare lapping edge. The specimen shall be prepared for testing by covering the bare lapping edge of the one strip with 1.5 g. of cement, spreading it

[3] 1944 Book of A.S.T.M. Standards, Part I.

[4] See the Standard Method of Test for Flash Point by Means of the Tag Closed Tester (A.S.T.M. Designation: D 56), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

evenly, and then allowing the strip to remain undisturbed for 10 min. at a temperature of 70 to 90 F. (21 to 32 C.). The reverse side of the other strip shall then be placed upon the cement-coated lapping edge in such a manner as to form a complete specimen 9 in. in length by 1 in. in width that includes a regular 3-in. cemented lap. A 50-lb. weight shall be placed upon the cemented lap and allowed to remain for 2 min. while the specimen is maintained at a temperature of 70 to 90 F. (21 to 32 C.). The specimen shall then be maintained at the temperature and for the period prescribed in Table II, and tested immediately in a tension testing machine[5] operated at a speed of head of 12 ± 2 in. per min.

TABLE II.—REQUIREMENTS FOR STRENGTH-OF-LAP TEST.

Age of Cemented Lap	Aging Temperature	Tensile Strength of Lap, min., lb.
30 min.	77 F. (25 C.)	6
60 min.	77 F. (25 C.)	8
24 hr.	77 F. (25 C.)	17
24 hr.	20 F. (−6.7 C.)	30
24 hr.	100 F. (38 C.)	18

There shall be furnished with each roll of one "square" of roofing for concealed nail application not less than 2 pt. of lap-cement.

(*c*) *Special Fixtures.*—Special fixtures may be furnished in lieu of nails and cement, if agreed upon by the purchaser and the seller.

Sampling and Testing

13. The material shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the Tentative Methods of Testing Asphalt Roll Roofing, Cap Sheets, and Shingles (A.S.T.M. Designation: D 228) of the American Society for Testing Materials.[6]

Inspection

14. Inspection of material shall be made as agreed upon by the purchaser and the seller as part of the purchase contract.

Basis of Rejection

15. Failure to conform to any one of the requirements prescribed in these specifications shall constitute grounds for rejection. In case of rejection, the seller shall have the right to reinspect the rejected shipment and resubmit the lot after removal of those packages not conforming to the specified requirements.

[5] A Scott Tester or equivalent tension testing machine is suitable for this purpose.

[6] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Tentative Specifications for

ASPHALT ROOFING SURFACED WITH POWDERED TALC OR MICA[1]

A.S.T.M. Designation: D 224 – 44 T

ISSUED, 1933; REVISED, 1934, 1935, 1937, 1941, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These specifications cover asphalt roofing in sheet form surfaced with finely powdered mineral matter, either 36 or 32 in. in width, composed of roofing felt saturated and coated on both sides with asphalt.

Manufacture

2. In the process of manufacture, a single thickness of dry roofing felt shall be impregnated with a hot asphaltic saturant, then coated on both sides with a hot asphaltic coating compounded with a finely powdered mineral filler, substantially insoluble in water, and finally surfaced on both sides with powdered talc or mica to prevent the roofing from sticking in the package.

Character of Felt

3. The felt shall be a roofing felt produced by "felting" vegetable or animal fibers or mixtures thereof. The surface of the felt shall be uniformly smooth. Upon splitting or tearing on the bias, the felt shall appear reasonably free from lumps of underbeaten stock and particles of foreign substances (that is, fragments of stone, metal, leather, rubber, etc.).

Character of Saturant and Coatings

4. The bitumen of the saturant and coatings shall be composed of asphaltic materials.

Character of Saturated Felt

5. The felt shall be completely and uniformly saturated, and shall show no unsaturated spots at any point upon cutting 2-in. strips at random across the entire sheet and splitting them open for their full length.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] Latest revision accepted by Committee E-10 on Standards, August 28, 1944.

Prior to their present publication as tentative, these specifications were published as tentative from 1925 to 1927, being revised in 1926 and 1927. They were adopted in 1927, published as standard from 1927 to 1933, but withdrawn and republished as tentative in 1933.

Emergency Alternate Provisions EA – D 224b applying to this standard appear in the pink section of this book.

Surface Finish

6. (*a*) The surface shall be smooth or veined in appearance.

(*b*) The coating and surfacing shall be applied uniformly and of approximately equal thickness on both sides and up to the edges of the sheet.

Physical Properties

7. The fabricated product shall conform to the requirements prescribed in Table I.

TABLE I.—PHYSICAL REQUIREMENTS OF ASPHALT ROOFING.

Width	The roofing shall be put up in rolls and shall be either 36 or 32 in. in width, as may be specified, within a permissible variation of ±¼ in.			
Area	The average area of the rolls examined shall contain not less than 108 or 216 sq. ft., as specified, which shall be sufficient to cover 100 or 200 sq. ft., respectively, of roof surface.			
Pliability at 77 F. (25 C.)	At least 8 strips out of 10 shall not crack when bent 90 deg. at a uniform speed over a rounded corner of ½-in. radius.			
Behavior on heating to 176 F. (80 C.) for 2 hr	Not more than 1.5 per cent volatile matter loss. There shall be no flowing, sagging, blistering, or absorption of the asphalt coatings.			
	65-lb. Grade		55-lb. Grade	
	Max., lb.	Min., lb.	Max., lb.	Min., lb.
Weight per roll, net average per 108 sq. ft.	....	61.0	....	51.0
Weight of dry felt per 108 sq. ft.	....	13.05	....	10.8
Weight of saturant (soluble in CS_2) per 108 sq. ft.[a]	....	20.9	....	17.3
Weight of mineral filled coating and surface mineral matter per 108 sq. ft.	....	18.0	....	18.0
Weight of any 108 sq. ft. roll of complete roofing in the shipment	....	60.0	....	50.0
Percentage by weight of mineral matter passing a No. 100 (149-micron) sieve on basis of total weight of mineral filled coating and surface mineral matter	50.0 per cent	15.0 per cent	50.0 per cent	15.0 per cent

[a] The weight of saturant per 108 sq. ft. shall be not less than 1.6 times the weight of the dry felt.

Freedom from Defects

8. The finished material shall be free of visible external defects, such as holes, ragged or untrue edges, breaks, cracks, tears, protuberances, and indentations.

Pliability and Stickiness

9. The finished product shall not crack nor be so sticky as to cause tearing or material damage upon being unrolled at atmospheric temperatures above 50 F. (10 C.).

Packing

10. (*a*) The roofing shall be put up in rolls of either 108 or 216 sq. ft. No roll shall contain more than two pieces nor shall there be more than 3 per cent of rolls containing two pieces in any shipment. The rolls shall be tightly wound, and shall be securely wrapped in a substantial grade of paper completely encircling the roll and pasted at the overlap in such a manner as to prevent shifting from position. The ends of the rolls shall be protected with cloth covers or by other suitable means. The nails and lap-cement shall be packed in the core of each roll or furnished separately if so specified by the purchaser. If nails and lap cement are furnished separately, cloth covers or other end protection may be omitted from the ends of the rolls.

(*b*) Special packing shall be as agreed upon by the purchaser and the seller.

Marking

11. Each roll shall be plainly marked with the name and brand of the manufacturer.

Nails and Lap-Cement

12. (*a*) *Nails:* (*1*) *Dimensions.*—The gage of the wire shall be not greater

than Washburn & Moen No. 10 (0.135 in.) nor less than Washburn & Moen No. 12 (0.105 in.). The head shall be not less than $\frac{3}{8}$ in. in diameter, nor less than 0.025 in. in thickness. The shank shall be not less than $\frac{3}{4}$ in. in length and shall be smooth, barbed, or square and twisted (spiral). If barbed, the barbing shall not increase the diameter of the lower two thirds more than 0.01 in. The lower end of the shank shall be pointed. If square and twisted (spiral), the cross-sectional area shall be equivalent to that specified above.

(*2*) *Protective Coating.*—Nails shall be galvanized or sherardized, and shall resist four 1-min. dips in a copper sulfate solution when tested in accordance with the Standard Method of Test for Uniformity of Coating by the Preece Test (Copper Sulfate Dip) on Zinc-Coated (Galvanized) Iron or Steel Articles (A.S.T.M. Designation: A 239) of the American Society for Testing Materials.[3]

(*3*) *Quantity.*—There shall be furnished per "square" an average of not less than 252 nails for 36-in. roofing and not less than 275 nails for 32-in. roofing.

(*b*) *Lap-Cement.*—The cement shall be composed of bituminous materials dissolved in a volatile solvent, and shall be of such a nature as to firmly bind the laps without injurious effect upon the roofing. The cement shall have a minimum flash point of 71 F. (21.5 C.) when tested by the Tag closed-cup tester.[4] There shall be furnished for each "square" of 36-in. roofing not less than $\frac{3}{4}$ pt. of cement and for 32-in. roofing not less than 1 pt. of cement. The addition of 5 to 20 per cent by weight of short-fibered asbestos shall be permitted, in which case the quantity of cement shall be increased by $\frac{1}{4}$ pt. per "square."

(*c*) *Special Fixtures.*—Special fixtures may be furnished in lieu of nails and cement, if agreed upon by the purchaser and the seller.

Sampling and Testing

13. The material shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the Tentative Methods of Testing Asphalt Roll Roofing, Cap Sheets, and Shingles (A.S.T.M. Designation: D 228) of the American Society for Testing Materials.[5]

Inspection

14. Inspection of material shall be made as agreed upon by the purchaser and the seller as part of the purchase contract.

Basis of Rejection

15. Failure to conform to any one of the requirements prescribed in these specifications shall constitute grounds for rejection. In case of rejection, the seller shall have the right to reinspect the rejected shipment and resubmit the lot after removal of those packages not conforming to the specified requirements.

[3] 1944 Book of A.S.T.M. Standards, Part I.

[4] See the Standard Method of Test for Flash Point by Means of the Tag Closed Tester (A.S.T.M. Designation D 56), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

[5] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Tentative Specifications for

ASPHALT SHINGLES SURFACED WITH COARSE MINERAL GRANULES[1]

A.S.T.M. Designation: D 225 – 44 T

ISSUED, 1933; REVISED, 1934, 1935, 1937, 1941, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. (*a*) These specifications cover asphalt roofing in shingle form, composed of roofing felt saturated and coated on both sides with asphalt and surfaced on the weather side with granulated slate or equivalent mineral material (of solid or mixed colors as may be agreed upon by the purchaser and the seller), and on the reverse side with a surfacing material.

(*b*) Shingles supplied under these specifications[a] are intended to be used as follows with a "head lap"[b] of not less than 2 in.:

Style of Shingle	Exposure to Weather, in.	Type
Individual (American method)	4	I
	5	II
	5	III
Square tab strip	4	I
	5	II
	5	III
Hexagonal tab strip	4⅔	I
	5⅓	II
	5⅓	III

[a] Individual shingles and shingles of special shapes may also be furnished as agreed upon by the purchaser and the seller.

[b] Head lap is the shortest vertical distance between the top of the cut out or bottom edge of a course of shingles, and the most proximate underlying area of roof deck not covered by preceding courses of shingles.

Manufacture

2. In the process of manufacture, a single thickness of roofing felt shall be impregnated with a hot asphaltic saturant, then coated on both sides with at least one layer of a hot asphaltic coating compounded with a mineral filler substantially insoluble in water, and finally completely surfaced on the weather side with mineral granules embedded in the hot asphaltic coating. The reverse side shall be covered with a suitable material to prevent the shingles from sticking together in the package. For type III shingles the thick portion of the shingles shall extend at least 1 in. on the unexposed portion to provide for proper nailing.

Character of Felt

3. The surface of the felt shall be uniformly smooth. Upon splitting or tearing on the bias, the felt shall appear

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] Latest revision accepted by Committee E-10 on Standards, August 28, 1944.

Prior to their present publication as tentative, these specifications were published as tentative from 1925 to 1927, being revised in 1926 and 1927. They were adopted in 1927, published as standard from 1927 to 1933, being revised in 1929, but withdrawn and republished as tentative in 1933.

reasonably free from lumps of underbeaten stock and particles of foreign substances (that is, fragments of stone, metal, leather, rubber, etc.).

Character of Saturant and Coatings

4. The bitumen of the saturant and coatings shall be composed of asphaltic materials.

Character of Saturated Felt

5. The felt shall be completely and uniformly saturated and shall show no unsaturated spots at any point upon cutting strips 2 in. in width across the entire shingle and splitting the strips open for their entire length.

Surface Finish

6. (*a*) The weather surface shall be uniform in finish and texture and may be embossed to simulate a grain texture. The mineral granules shall cover the entire surface and shall be firmly embedded in the asphalt coating.

(*b*) The reverse side shall have a coating of asphalt and shall be surfaced with suitable material to prevent the shingles from sticking in the package. The reverse side may be surfaced with granular material.

Physical Properties

7. The fabricated product shall conform to the requirements prescribed in Table I.

Freedom from Defects

8. The finished shingles shall be free from visible external defects, such as holes, ragged or untrue edges, rents, cracks, indentations and lumps of coating.

Stickiness

9. The shingles shall not stick together in the package so as to cause damage upon being unpacked when at atmospheric temperatures above 50 F. (10 C.).

Packing

10. The shingles shall be packed in

TABLE I.—PHYSICAL REQUIREMENTS OF ASPHALT SHINGLES.

Shape	The form and size of the shingles shall be as agreed upon by the purchaser and the seller.					
Area	Sufficient material per "square" shall be furnished to cover adequately 100 sq. ft. of roof surface.					
Behavior on heating to 176 F. (80 C.) for 2 hr	Not more than 1.5 per cent volatile matter loss. The granular surfacing shall not slide more than $\frac{1}{16}$ in. when suspended vertically.					
	Type I		Type II		Type III	
	Max., lb.	Min., lb.	Max., lb.	Min., lb.	Max., lb.	Min., lb.
Weight, net average of finished shingles per 108 sq. ft.[a]	.....	86.0	.....	97.2	.....	89.5
Weight of roofing per 108 sq. ft. (individual package)	.....	83.0	.....	94.0	.....	86.5
Weight of exposed area per 108 sq. ft.	.....	83.0	.....	94.0	.....	108.0
Weight of dry felt per 108 sq. ft.	.....	10.8	.....	13.5	.....	10.8
Weight of saturant per 108 sq. ft.[b]	.....	18.9	.....	23.6	.....	18.9
Weight of weather side mineral filled coating per 108 sq. ft.	35.0	16.0	46.0	16.0	60.0[c]	20.0[c]
Weight of mineral matter per 108 sq. ft. passing No. 6 (3360-micron) and retained on No. 100 (149-micron) sieve	.....	20.0	.....	20.0	.....	20.0[c]
Percentage by weight of mineral matter passing No. 100 (149-micron) sieve on basis of the sum of the bitumen in the coatings and mineral matter passing No. 100 (149-micron) sieve	50.0	15.0	50.0	15.0	50.0	15.0
Mineral matter (including surfacing and filler), per cent of total weight of finished shingle	.....	.....	.....	.....	.....	35.0

[a] This shall be calculated as follows: $\frac{\text{net weight of packages inspected}}{\text{sq. ft. area per shingle} \times \text{number of shingles}} \times 108.$

[b] The weight of saturant per 108 sq. ft. shall be not less than 1.75 times the weight of the dry felt.

[c] Exposed area only.

cardboard cartons or between wood or cardboard, or both, and tied securely so as to make a package, weighing not more than 135 lb. gross.

Marking

11. Each package shall be plainly marked with the name and brand of the manufacturer, as well as the style, type, and color of product. Direction sheets for application shall be included in at least every fifth package.

Sampling and Methods of Test

12. The material shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the Tentative Methods of Testing Asphalt Roll Roofing, Cap Sheets, and Shingles (A.S.T.M. Designation: D 228).[3]

Inspection

13. Inspection of material shall be made as agreed upon by the purchaser and the seller as part of the purchase contract.

Basis of Rejection

14. Failure to conform to any one of the requirements prescribed in these specifications shall constitute grounds for rejection. In case of rejection the seller shall have the right to reinspect the rejected shipment and resubmit the lot after removal of those packages not conforming to the specified requirements.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Tentative Methods of

TESTING FELTED AND WOVEN FABRICS SATURATED WITH BITUMINOUS SUBSTANCES FOR USE IN WATERPROOFING AND ROOFING[1]

A.S.T.M. Designation: D 146 – 44 T

ISSUED, 1938; REVISED, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These methods cover the examination of felted or woven fabrics saturated (but not coated) with asphalt or coal-tar materials, for use in the "membrane" system of waterproofing or for the construction of built-up roof coverings.

Sampling

2. From each shipment or fraction thereof representing a product of the same kind, class, and weight, a number of rolls shall be selected at random, equivalent to one-half the cube root of the total number of rolls in the lot, except that in lots of 1000 or less, five rolls shall be taken. If the cube root, as calculated, proves to be a fractional number, it shall be expressed as the next higher whole number. For convenience, the following table is given, showing the number of rolls to be selected from shipments of various sizes:

Packages in Shipment	Number of Packages Selected
Up to 1 000	5
1 001 to 1 728	6
1 729 to 2 744	7
2 745 to 4 096	8
4 097 to 5 832	9
5 833 to 8 000	10
8 001 to 10 648	11
10 649 to 13 842	12
13 843 to 17 576	13
17 577 to 21 952	14

EXAMINATION OF FINISHED FABRIC

Width

3. The width of each roll selected shall be measured to the nearest $\frac{1}{32}$ in. The minimum and maximum width so obtained shall be reported.

Gross Weight per Roll

4. Each roll selected shall be weighed intact to the nearest $\frac{1}{4}$ lb. and the minimum and maximum gross weight shall be reported.

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] Prior to their present publication as tentative, these methods were published as tentative from 1922 to 1927, being revised in 1923, 1925, 1926, and 1927. They were adopted in 1927, published as standard from 1927 to 1938, being revised in 1937, but withdrawn and republished as tentative in 1938.

Weight of Wrapping and Packing Materials

5. Each roll selected shall be stripped. All the wrappers and packing material shall be weighed together to the nearest $\frac{1}{4}$ lb. and the average maximum weight thereof per roll shall be reported.

Area per Roll

6. Each roll selected shall be unwound, observing the workmanship and finish while so doing. The width and length of each roll selected shall then be measured to the nearest $\frac{1}{4}$ in. and the area of material contained in each roll shall be calculated in square feet.

Net Weight per Roll

7. The rolls shall be rewound, fastened with pieces of light string, and then each roll shall be weighed to the nearest $\frac{1}{4}$ lb. In the case of felted fabrics, the weight of each roll in pounds per 100 sq. ft. shall be calculated and the maximum and minimum weights reported. The average weight for the rolls sampled, which shall be regarded as the corresponding weights of the entire lot, shall also be reported. In the case of woven fabrics, the weight of each roll shall be calculated in ounces per square yard, and the minimum weight reported, likewise recording the average for the rolls sampled, which shall be regarded as the corresponding weights of the entire lot.

Weight per Unit Area and Selecting Representative Sample

8. From the rolls examined, the one whose weight per unit area is nearest the average weight per unit area of the lot shall be selected. The roll so selected shall be laid flat, the first convolution or two carefully unwound, and with a knife and straight edge the sheet shall be cleanly cut across at right angles to the edges. A sample measuring exactly 30 in. in the direction of the roll's length shall be removed. If the material is surfaced with sand or other fine material, any detached particles belonging to each 30-in. sample shall be retained. The width of each sample shall be measured to the nearest $\frac{1}{32}$ in. Each sample, together with any detached surfacing, shall be weighed to the nearest gram. The weight in pounds per 100 sq. ft. or the weight in ounces per square yard shall be calculated as follows:

$$\text{Weight, lb. per 100 sq. ft.} = 1.0582 \times \frac{A}{B}$$

$$\text{Weight, oz. per sq. yd.} = 1.5238 \times \frac{A}{B}$$

where:

A = weight of 30-in. sample in grams, and

B = width of 30-in. sample in inches.

This weight so determined shall check within 1 per cent the average weight per unit area of the lot (Section 7). If the sample tested fails to conform to this requirement, then additional samples shall be cut from the same roll, until one is obtained which does, and this sample shall be reserved for further examination as described in Sections 9 to 16.

NOTE.—As a referee method or in case any dispute arises regarding the properties as may be ascertained from the particular sample selected, a 30-in. sample shall be taken from each roll selected in accordance with Section 2 and examined separately.

Detached Comminuted Surfacing

9. If the material is surfaced with sand or other finely comminuted material, the surfacing shall be swept with a moderately stiff brush from all the 30-in. samples selected in accordance with Section 8. All the material thus removed shall be caught and weighed to the nearest ounce. From the aggregate areas of all the sections taken, the average weight of detached comminuted

surfacing shall be calculated in pounds per 100 sq. ft.

Thickness

10. The thickness of each sample shall be measured at ten equally spaced points at least 1 in. from a cut edge with a dead-weight micrometer gage having a cylindrical foot and anvil with flat bearing surfaces 1 sq. in. in area. The load used shall be 2 psi. The thickness for each roll sampled shall be averaged and the minimum, maximum, and average thicknesses calculated to the nearest 0.001 in.

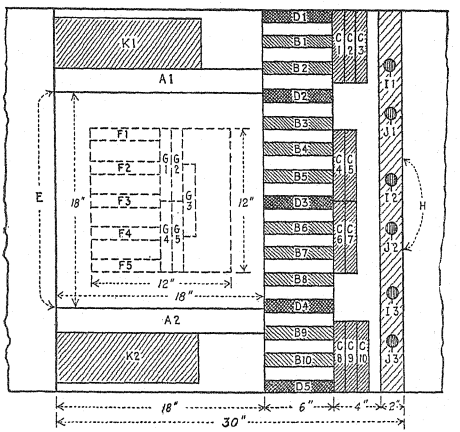


FIG. 1.—Location of Test Specimens in Sample.

Moisture

11. From each sample, 2-in. test specimens shall be cut as shown at *A-1* and *A-2* in Fig. 1. The 2-in. specimens shall be cut into 1-in. squares. About 50 g., selected at random, shall be accurately weighed and distilled with 100 ml. of a coal-tar distillate of which 5 to 10 per cent boils below 100 C. (212 F.) and of which at least 90 per cent distills up to 180 C. (356 F.) in an apparatus of the form and under the conditions prescribed in the Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (A.S.T.M. Designation: D 95) of the American Society for Testing Materials.[3] The flask containing the solvent and sample shall be heated in a paraffin bath at a temperature of 302 to 338 F. (150 to 170 C.) until no further water passes over. The distillate shall be allowed to stand in the graduated receiver until the water separates, whereupon the volume of water shall be measured and its weight calculated. From this, the average percentage of moisture in the fabric, as received, shall be calculated.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Strength

12. (*a*) *Felted Fabrics.*—With a photographic trimmer having a stop attached exactly 1 in. behind the blade, ten test specimens from the sample shall be cut with the fiber grain, as shown at *B-1* to *B-10* (Fig. 1), and ten strips across the fiber grain as shown at *C-1* to *C-10* (Fig. 1). Each specimen shall measure 6 in. and have parallel sides 1 in. apart (within $\frac{1}{32}$ in.) with the edges cut straight and clean. Both sets of specimens shall be tested at 77 F. (25 C.), using a tension testing machine such as the Scott Strength Tester (type F, 100-lb. capacity), the Perkins Strip Tester, or equivalent apparatus, in which the clamps are attached to swivels free to move in any direction. The test specimens shall be gripped $1\frac{1}{2}$ in. on each end, leaving approximately 3 in. between the clamps. The tension shall be increased by causing the lower clamp of the machine to travel at a uniform speed of 12 in. per min. If any specimen breaks nearer than $\frac{1}{2}$ in. to either clamp, the reading shall be disregarded, and an additional specimen shall be tested in its place. The ten readings, with and across the grain, respectively, shall be averaged for each roll sampled. From these results the average strength with and across the fiber grain, respectively, of the fabric as supplied, shall be calculated.

(*b*) *Woven Fabrics.*—Woven fabrics shall be tested at 70 F. (21.1 C.) in accordance with the grab method as described in Section 10 of the Standard General Methods of Testing Woven Textile Fabrics (A.S.T.M. Designation: D 39) of the American Society for Testing Materials.[4]

NOTE.—As a referee method, or in case any dispute arises regarding the strength, the test shall be repeated, with the exception that the fabric before being tested shall be exposed at least 2 hr. in an atmosphere of 65 per cent relative humidity at 70 F. (21.1 C.).

Pliability

13. (*a*) *Felted Fabrics.*—From the sample selected in accordance with Section 8, ten test specimens 1 in. in width by 8 in. in length shall be cut from the sample, five in the direction of and five across the fiber grain. They shall be immersed in water at 77 F. (25 C.) for 10 to 15 min., then removed, and each specimen immediately bent 90 deg. over the rounded edge of a block at a uniform speed in approximately 2 sec. The block shall be 3-in. square by 2 in. in thickness with rounded corners of $\frac{1}{2}$-in. radius for 15-lb. felts and $\frac{3}{4}$-in. radius for 30-lb. felts. In bending, the specimen shall be held by hand tightly against the upper 2-in. face of the block, and the projecting end of the specimen shall be bent over the rounded corner without exerting any strain other than that required to keep the specimen in contact with the block and to avoid kinking. Any surface ruptures exceeding $\frac{1}{8}$-in. in length shall be considered failures.

(*b*) *Woven Fabrics.*—From the sample selected in accordance with Section 8, five test specimens 1 in. in width by 6 in. in length shall be cut in the direction of the warp. They shall be immersed in a cooling mixture of ice and water at 32 F. (0 C.) for 10 to 15 min., then removed and each specimen immediately bent over a $\frac{1}{16}$-in. mandrel through an arc of 180 deg. at a uniform speed in approximately 2 sec. and then through 360 deg. over the same mandrel in the opposite direction. The specimens shall then be dried thoroughly and examined. If one or more of the test specimens crack, ten specimens from another portion of the sample shall be cut and the test repeated. If one or more of these specimens crack,

[4] 1944 Book of A.S.T.M. Standards, Part III.

the material shall be considered as failing to conform to the specifications.

Water Absorption

14. (*a*) A specimen 18 in. square (as shown at *E*, Fig. 1) shall be cut from each sample, weighed, and completely immersed in distilled water at 77 F. (25 C.) for 24 hr. The specimen shall then be removed and dried superficially by pressing lightly between two towels. As the moisture enters through the edges of the sheet more rapidly than through the surfaces, each specimen shall be trimmed to exactly 12 in. square, representing four-ninths of the original area, and reweighed rapidly. The increase in weight shall be calculated on the basis of the original test specimen by multiplying by $2\frac{1}{4}$. The percentage increase in weight represents the water absorption. The minimum, maximum, and average for the lot shall be calculated.

(*b*) Five test specimens shall be cut with the fiber grain as shown at *F-1* to *F-5* (Fig. 1); five other specimens shall be cut across the fiber grain as shown at *G-1* to *G-5* (Fig. 1); and the strength shall be redetermined as described in Section 12. The percentage decrease in strength after the specimen has been subjected to water shall be calculated.

Loss on Heating of Asphalt Saturated Fabrics

15. From each sample, 12 by 6-in. test specimens shall be cut at *K-1* and *K-2* (Fig. 1), care being taken not to disturb any of the detached surfacing. Each specimen shall be weighed and suspended in the center of an air oven maintained at 221 ± 5 F. (105 ± 3 C.) by means of a thin wire fastened through holes punctured near one edge. The thermometer shall be inserted in the oven to such a depth that its bulb will be in line with the center of the specimens. The specimens shall be kept in the oven for exactly 5 hr., then cooled and removed carefully, and each specimen weighed. The average loss shall be calculated as a percentage and the average percentage of moisture (determined in Section 11) deducted. The final figure shall represent the average loss on heating, exclusive of moisture.

EXAMINATION OF DESATURATED FABRIC

Weight of Desaturated Fabric

16. From each roll selected a 2-in. strip (within $\frac{1}{32}$ in.) shall be cut across the specimen as shown at *H* (Fig. 1). Each strip shall be extracted with carbon disulfide[5] in an extractor described in Section 3(*b*) of the Standard Methods of Testing Bituminous Mastics, Grouts, and Like Mixtures (A.S.T.M. Designation: D 147) of the American Society for Testing Materials,[3] the extraction being continued for several hours after the drippings have become colorless. The desaturated fabric shall be removed and heated in a ventilated oven at 225 F. (107.1 C.), cooled in a desiccator, any adhering comminuted surfacing being brushed off and retained, and weighed as rapidly as possible. The heating shall be repeated until the weight of the fabric remains constant as determined by two consecutive weighings, taken not less than 10 min. apart, which shall show a further loss of not more than 0.1 per cent. This will give the weight of the fabric in the moisture-free state. Where a coal-tar pitch saturant has been used, the moisture-free weight of the desaturated fabric shall be corrected for the carbonaceous matter retained mechanically in its interstices, by means of the method described in Section 17.

Retained Carbonaceous Matter

17. The following colorimetric method shall be used to determine the carbona-

[5] In this test, chloroform may be used as an equivalent solvent.

ceous matter derived from a coal-tar pitch saturant and retained by the desaturated fabric:

(*a*) About 15 g. of an unsaturated fabric of the same general character as the one under examination, shall be macerated by boiling in water, disintegrating with a rotary egg-beater and picking the fibers apart with needles. The fibers shall be filtered through fine cloth and dried to constant weight at 225 F. (107.1 C.). A 1-g. portion of the fibers shall be accurately weighed into a flask and diluted to exactly 100 ml. with distilled water at room temperature. About 50 g. of glass beads shall be added and the contents of the flask shaken vigorously until the fibers are reduced to a homogeneous pulp in uniform suspension.

(*b*) A distilled coal tar, having approximately 10 to 25 per cent of insoluble carbonaceous matter, shall be procured. The carbonaceous matter shall be extracted with benzol until it is free from soluble matter, then dried to constant weight at 225 F. (107.1 C.). A 1-g. portion of the purified carbonaceous matter shall be accurately weighed and diluted to exactly 100 ml. at room temperature with a starch solution of a consistency sufficient to carry the carbonaceous matter into temporary suspension. (A 12.5 per cent solution is recommended.)

(*c*) The liquid carrying the fibers, obtained as described in Paragraph (*a*), shall be titrated with the suspension of carbonaceous matter, obtained as described in Paragraph (*b*), and from time to time a field prepared from a drop of the well agitated mixture shall be examined under a microscope at 100 diameters magnification until the color exactly matches a field prepared from the desaturated fabric under examination (obtained as described in Section 16), when both are viewed side by side under identical conditions. The end point is fairly sharply defined. The burette reading gives directly the percentage of carbonaceous matter adhering to the moisture-free fabric under examination.

(*d*) The weight of moisture-free fabric determined in Section 16 shall be corrected by deducting the weight of adhering carbonaceous matter.

Total Comminuted Surfacing

18. The total comminuted surfacing (plus any filler present in the bituminous saturant) represents the sum of the detached comminuted surfacing (Section 9), and the amount brushed off from the surface of the desaturated fabric (Section 16), plus the amount recovered upon evaporating and igniting the bituminous extract obtained in accordance with Section 16.

Bituminous Saturant

19. The weight of bituminous saturant represents the difference between the weight of the original fabric (Section 7), and the sum of the weights of the moisture-free desaturated fabric (Sections 16 and 17), plus moisture (Section 11), plus the total comminuted surfacing and filler present (Section 18).

Weight

20. (*a*) *Woven Fabrics.*—Woven fabrics shall be tested for weight and number of threads per inch in accordance with Sections 6 and 7 of the Standard General Methods of Testing Woven Textile Fabrics (A.S.T.M. Designation: D 39) of the American Society for Testing Materials.[4]

(*b*) *Felted Fabrics.*—The weight in pounds per 480 sq. ft. of the desaturated felt (corrected for any carbonaceous matter present) for each specimen examined shall be calculated separately as follows:

$$\text{Number} = \frac{\text{wt. of specimen in grams}}{\text{area of specimen in sq. cm.}} \times 983$$

or

$$\text{Number} = \frac{\text{wt. of specimen in grams}}{\text{area of specimen in sq. in.}} \times 152$$

This represents the "number" of the moisture-free felt on the felt makers' scale. The minimum, maximum, and average number of the felt in its moisture-free state shall be calculated. This may be corrected to the condition in which it actually existed in the material under examination, by adding thereto the percentage of moisture as determined in Section 11.

Ash

21. A representative sample shall be secured by cutting from each specimen of desaturated felt a piece about $\frac{1}{2}$ in. in diameter as shown at *I-1*, *I-2*, and *I-3* (Fig 1). About 25 g. selected at random from all the specimens sampled in this manner shall be accurately weighed, and incinerated in a weighed porcelain or quartz crucible either over an open flame or in a muffle, until all the carbon is consumed. A few drops of ammonium carbonate solution shall be added, the sample ignited gently, and weighed. The percentage of ash shall be calculated on the basis of the moisture-free felt.

NOTE.—As a referee method, or in case greater accuracy is desired, the three portions taken from each specimen roll shall be weighed and ignited separately. The minimum, maximum, and average amount of ash present shall be calculated on the basis of the moisture-free felt.

Tentative Methods of

TESTING ASPHALT ROLL ROOFING, CAP SHEETS, AND SHINGLES[1]

A.S.T.M. Designation: D 228 – 44 T

ISSUED, 1930; REVISED, 1931, 1933, 1937, 1942, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These methods cover the procedures for the physical testing and chemical examination of roofing and shingles composed of asphalt-saturated roofing felt coated to various extents with asphalt and the coated portion surfaced with mineral powders or granules.

Types of Roofing and Shingles

2. Asphalt roll roofings, cap sheets, and shingles may be divided into three types (see Fig. 1), as follows:

Type X.—A single thickness of asphalt-saturated felt, coated with asphalt compounded with a finely powdered mineral filler and surfaced with powdered mineral matter such as talc or mica.

NOTE.—Type X is commercially known as "smooth-surfaced roll roofing" and is covered by the Tentative Specifications for Asphalt Roofing Surfaced with Powdered Talc or Mica (A.S.T.M. Designation: D 224) of the American Society for Testing Materials.[3]

Type Y.—Similar to type X, but with the asphalt coating on the weather side surfaced with coarse granules.

NOTE.—When coated with coarse mineral granules, type Y is commercially known as "mineral-surfaced roll roofing" and is covered by the Tentative Specifications for Asphalt Roofing Surfaced with Coarse Mineral Granules (A.S.T.M. Designation: D 249);[3] or when cut into slabs or shingles it is commercially known as "asphalt shingles" and is covered by the Tentative Specifications for Asphalt Shingles Surfaced with Coarse Mineral Granules (A.S.T.M. Designation: D 225)[3] of the American Society for Testing Materials. The reverse side usually is surfaced with powdered talc or mica.

Type Z.—A single thickness of asphalt-saturated roofing felt, coated on the weather side for approximately one

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] Latest revision accepted by Committee E-10 on Standards, August 28, 1944.

Prior to their present publication as tentative, these methods were published as tentative from 1925 to 1927, being revised in 1926 and 1927. They were adopted in 1927, published as standard from 1927 to 1930, but withdrawn and republished as tentative in 1930.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

half of its width with asphalt compounded with fine mineral matter, and the coated portion surfaced with coarse mineral granules.

NOTE.—Type Z is commercially known as "mineral-surfaced cap sheet" and is covered by the Standard Specifications for Wide Selvage Asphalt Roofing Surfaced with Coarse Mineral Granules (A.S.T.M. Designation: D 371) of the American Society for Testing Materials.[3]

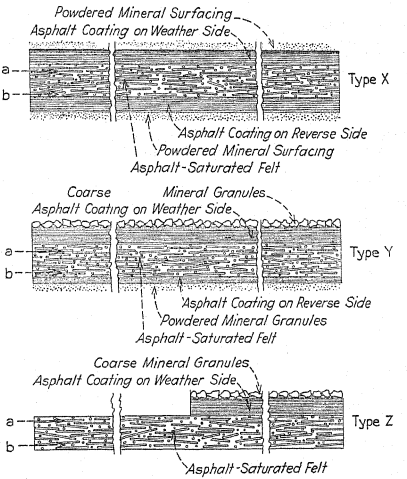


FIG. 1.—Construction of Asphalt Roll-Roofing, Cap Sheets, and Shingles.

Sampling

3. From each shipment or fraction thereof representing a product of the same kind, class, and weight, a number of rolls or bundles shall be selected at random, equivalent to one half the cube root of the total number of rolls or bundles included in the lot, except that in lots of 1000 or less five rolls or bundles shall be taken. If the cube root, as calculated, proves to be a fractional number, it shall be expressed as the next higher whole number. For convenience, the following table is given, showing the number of rolls or bundles to be selected from shipments of various sizes:

Packages in Shipment	Number of Packages Selected
Up to 1 000	5
1 001 to 1 728	6
1 729 to 2 744	7
2 745 to 4 096	8
4 097 to 5 832	9
5 833 to 8 000	10
8 001 to 10 648	11
10 649 to 13 824	12
13 825 to 17 576	13
17 577 to 21 952	14

METHODS OF PHYSICAL TESTING

Asphalt Roll Roofing and Cap Sheets

Weight of Roll, and Packing Material, Nails, Cement

4. Each roll selected shall be weighed

intact to the nearest $\frac{1}{4}$ lb. The wrappers, packing material, nails, and cement from each roll selected shall be weighed together to the nearest $\frac{1}{4}$ lb. and the average weight thereof per "square" shall be recorded. The quantity and character of nails and cement shall be recorded.

Weight of Loose Surfacing

5. Each roll selected shall be unwound and the workmanship and finish of the roofing observed. If any loose surfacing is noted, it shall be shaken off, the roll reweighed, and the loss of weight recorded.

Width, Length, and Area

6. The width and length of each roll selected shall be measured to the nearest $\frac{1}{2}$ in., and the area of material contained in each roll shall be calculated in square feet. The average area of the rolls examined shall be calculated from these values.

Minimum Weight per 108 sq. ft.

7. From the information obtained under Sections 5 and 6, the net weight, exclusive of loose surfacing, of roofing per 108 sq. ft. contained in each roll selected shall be calculated. The minimum weight per 108 sq. ft. of the rolls examined shall be recorded. This shall be regarded as the minimum weight per 108 sq. ft. of the lot.

Average Weight per 108 sq. ft.

8. The average weight per 108 sq. ft. for the rolls examined shall be calculated. This shall be regarded as the average weight per 108 sq. ft. of the lot.

Weight per Unit Area and Selecting Representative Sample

9. From the rolls examined, the one whose weight per 108 sq. ft. is nearest the average weight of the lot shall be selected. The roll so selected shall be laid flat, the first convolution or two carefully unwound, and with a knife and straightedge the sheet shall be cleanly cut across at right angles to the edges. A sample measuring $30 \pm \frac{1}{32}$ in. in the direction of the roll's length shall be removed. Any unsurfaced lapping edge of the roll shall be removed and the width of the surfaced area shall be measured to the nearest $\frac{1}{32}$ in. The weight of the sample in ounces shall be determined, neglecting any loose surfacing. The weight in pounds per 108 sq. ft. shall be calculated as follows:

$$\text{Weight, lb. per 108 sq. ft.} = \frac{A}{B} \times 32.4$$

where:

A = weight of 30-in. sample in ounces,
B = width of 30-in. sample in inches, and
32.4 = factor for converting ounces per measured unit area (30 in. $\times$ B) to pounds per 108 sq. ft.

The weight so determined shall check within 1.5 per cent the average weight per 108 sq. ft. of the shipment (Section 8). If the sample tested fails to conform to this requirement, then additional samples shall be cut from the same roll, until one is obtained which does, and this sample shall be reserved for further examination as described in Sections 16 to 25.

NOTE.—As a referee method, or in case any dispute arises regarding the properties as may be ascertained from the particular sample tested, a 30-in. sample shall be taken from each roll selected in accordance with Section 3 and examined separately.

Asphalt Shingles

Weight of Packing Material

10. Each bundle selected shall be weighed to the nearest $\frac{1}{4}$ lb. The packing materials shall be weighed to the

nearest $\frac{1}{4}$ lb. and the weight recorded separately for each bundle. The average weight of packing material per "square" shall be recorded.

Weight of Loose Surfacing

11. The shingles in each bundle, *a*, shall be counted and the workmanship and finish of the shingles shall be observed. If any loose surfacing is noted, it shall be shaken off, the bundle reweighed, and the loss of weight recorded.

Net Weight and Area

12. From the information obtained under Sections 10 and 11, the net weight, *b*, of each bundle selected, exclusive of loose surfacing, shall be calculated. The dimensions of two representative shingles shall be accurately measured and the net area, *c*, of material in each shingle calculated in square feet.

Minimum Weight per 108 sq. ft.

13. The area in square feet of material contained in each bundle selected shall be calculated ($a \times c$). The net weight of roofing material per 108 sq. ft. contained in each bundle shall be calculated $\left(\frac{b}{a \times c} \times 108\right)$, and the minimum weight per 108 sq. ft. recorded. This shall be regarded as the minimum weight per 108 sq. ft. of the lot.

Average Weight per 108 sq. ft.

14. The average weight, *d*, per 108 sq. ft. for the bundles examined shall be calculated. This shall be regarded as the average weight per 108 sq. ft. of the lot.

The average weight per shingle shall be calculated $\left(\frac{c \times d}{108}\right)$.

Selecting Representative Sample

15. From the bundles selected, a definite number of the representative shingles whose weight per 108 sq. ft. falls within 1.5 per cent of the average weight of the lot (Section 14) shall be reserved for further examination in accordance with Sections 17 to 26. The number of shingles so selected shall represent as closely as possible 6 sq. ft. of material, and shall be selected as far as possible from different bundles.

NOTE.—As a referee method, or in case any dispute arises regarding the properties as may be ascertained from the particular sample tested, then a similar sample shall be taken from each bundle of shingles selected in accordance with Section 3 and examined separately.

Asphalt Roll Roofing and Cap Sheets

Pliability

16. From the sample selected in accordance with Section 9, ten test specimens 1 in. in width by 8 in. in length shall be cut from the sample, five in the direction of and five across the fiber grain. They shall be immersed in water at 77 F. (25 C.) for 10 to 15 min., then removed, and each specimen immediately bent, with the weather side up, 90 deg. over the rounded edge of a block at a uniform speed in approximately 2 sec. The block shall be 3 in. square by 2 in. in thickness with rounded corners of $\frac{1}{2}$-in. radius for type X roofing and $\frac{3}{4}$ in. for types Y and Z roofing. In bending, the specimens shall be held by hand tightly against the upper 2-in. face of the block, and the projecting end of the specimen shall be bent over the rounded corner without exerting any strain other than that required to keep the specimen in contact with the block and to avoid kinking. Any surface ruptures exceeding $\frac{1}{8}$ in. in length shall be considered failures.

Asphalt Roll Roofing, Cap Sheets, and Shingles

Loss and Behavior on Heating

17. Two test specimens each approximately 4 by 4 in., shall be cut from the

sample selected in accordance with Section 9 in the case of asphalt roll roofing and cap sheets, and from that selected in accordance with Section 15 in the case of shingles. The specimens shall be weighed and then suspended vertically, in the same direction as the material would be applied to the roof, in the center of an air oven maintained at 176 ± 5 F. (80 ± 3 C.) by means of a thin wire fastened through holes punctured near one edge. The internal dimensions of the oven shall be not less than 12 by 12 by 12 in. An electrically-heated oven is recommended. The thermometer shall be inserted in the oven to such a depth that its bulb will be in line with the center of the specimens. The specimens shall be maintained at the prescribed temperature for exactly 2 hr., then cooled in a desiccator, and each specimen weighed. The average loss of volatile matter shall be calculated as a percentage. Any change in appearance of the specimen shall be recorded, such as flowing, sagging, blistering, or absorption of the asphalt coatings; also sliding of granular surfacing.

METHODS OF ANALYSIS

Analysis of Roofing Composition

18. The composition of the roofing shall be analyzed as follows:

(*a*) Weight of dry felt per 108 sq. ft.,

(*b*) Weight of saturant (soluble in carbon disulfide) per 108 sq. ft.,

(*c*) Weight of weather side coating (soluble in carbon disulfide) per 108 sq. ft. (which may be either side on Type X),

(*d*) Weight of reverse side coating (soluble in carbon disulfide) per 108 sq. ft. (which may be either side on Type X),

(*e*) Weight of mineral matter per 108 sq. ft. passing a No. 6 (3360-micron) sieve and retained on a No. 100 (149-micron) sieve, and

(*f*) Weight of mineral matter per 108 sq. ft. passing a No. 100 (149-micron) sieve.

Preparation of Specimens for Analysis

19. (*a*) From the sample representing the average weight of the lot of roofing, as obtained in Section 9 or 15, specimens shall be cut, each measuring approximately 2 in. in width by 8 in. in length. These specimens shall be weighed, the length and width measured to within $\frac{1}{32}$ in., and the weight calculated per 108 sq. ft. Any of the 2 by 8-in. specimens whose weight varies more than 1.5 per cent from the average weight of the lot as determined in Section 8 or 14 shall be rejected. This process shall be continued until eight acceptable specimens are obtained which shall be used for determining the composition of the roofing as described in Sections 20 to 26.

(*b*) Two of the weighed 2 by 8-in. specimens shall each be separated into three horizontal sections approximately at the points indicated by the arrows *a* and *b* in Fig. 1. (These strips may be cut to smaller size and into disks if so desired.) The asphalt coatings with attached mineral surfacings shall be removed in such a manner that some of the saturated felt adheres to them leaving a central section of saturated felt free from coating. Any mineral surfacing detached during this operation shall be added to the section to which it belongs.

Saturant in Dry Felt

20. The saturated central horizontal sections from the two test specimens (Section 19 (*b*)) shall be weighed and then extracted with carbon disulfide in a suitable extractor or centrifuge until the washings are colorless. (Benzol

having a boiling point of 80 to 82 C., carbon tetrachloride, or chloroform may be used if desired, but in case of dispute carbon disulfide shall be used.) The desaturated felt shall be dried in air, then placed in a tared weighing bottle and further dried at 105 to 110 C. (221 to 230 F.) for 30 min., and weighed. From this weight and the original weight of the saturated felt specimens, the percentage of asphalt saturant in the dry felt shall be calculated. This figure shall be used later for calculating the weight of saturant (soluble in carbon disulfide) per 108 sq. ft. The dry felt shall be preserved for the determination of ash (Section 23).

NOTE.—The saturant may be recovered by the method described in the Tentative Method of Test for Hot Extraction of Asphaltic Materials and Recovery of Bitumen by the Modified Abson Procedure (A.S.T.M. Designation: D 762).[3]

Weight of Weather Side Coating Soluble in Carbon Disulfide

21. (*a*) The horizontal sections of the weather side (which may be either side on type X roofing) containing the asphalt coating and mineral surfacing (Section 19 (*b*)) shall now be extracted with carbon disulfide (Note) in a suitable extractor until the washings are colorless. The insoluble material shall be removed and dried in air and the pieces of felt picked out and brushed free of adhering mineral matter. This felt shall then be placed in a tared weighing bottle, further dried at 105 to 110 C. (221 to 230 F.) for 30 min., and weighed. From this weight corrected for excess ash as in Section 23 and the percentage of asphalt saturant determined in Section 20, the weight of asphalt in this felt shall be calculated. The mineral matter shall be dried in air until free from carbon disulfide, then placed in a tared weighing bottle, further dried for 30 min. at 105 to 110 C. (221 to 230 F.), cooled in a desiccator, and weighed. This mineral matter shall be retained for sieve analysis (Section 25).

NOTE.—Carbon tetrachloride may be used in place of carbon disulfide, if desired. In case of dispute, carbon disulfide shall be used.

(*b*) *Calculation*—From the original weight of the sample subtract the combined weights of felt, asphalt, saturant in the felt, and mineral matter. The difference is the weight of weather side coating (soluble in carbon disulfide) per 108 sq. ft.

Weight of Reverse Side Coating Soluble in Carbon Disulfide

22. The horizontal sections of the reverse side (which on Type X roofing will be the side opposite to that selected under Section 21) containing the coating and mineral surfacing (Section 19 (*b*)) shall now be extracted with carbon disulfide (Note, Section 21 (*a*)) and the weight of coating soluble in carbon disulfide calculated as in Section 21. The mineral matter recovered shall be combined with that obtained from the weather side (Section 21) and retained for sieve analysis (Section 25). The felt shall be ashed as in Section 23, in order to determine the true weight of felt.

Ash of Desaturated Felt

23. The desaturated felt obtained in Sections 20, 21, and 22 from the center, and the weather and reverse sides shall be ashed separately in weighed crucibles, either over an open flame or in a muffle furnace, until all carbon has been consumed. The weight shall be determined. The percentage of ash in the center portion (Section 20) will be assumed to be the true percentage of ash of the entire felt. The excess ash in the weather side portion (Section 21) will be added to the mineral matter passing a No. 100 (149-micron) sieve as determined in Section 25. This weight

will also be used for correcting the total weight of desaturated felt and weight of saturant souble in carbon disulfide. In like manner, the excess ash in the reverse side portion will be added to the mineral matter passing a No. 100 sieve and used for correcting the total weights of felt and saturant soluble in carbon disulfide.

Weight of Dry Desaturated Felt per 108 sq. ft.

24. The combined weights of the felt from the center portion obtained in Section 20 and the weights of the felts from the weather side and reverse side portions obtained in Sections 21 and 22, respectively, corrected for excess ash (Section 23) shall be the total weight of dry felt in the two 2 by 8-in. specimens. From this weight calculate the weight of dry felt per 108 sq. ft.

Weight of Mineral Matter

25. (*a*) The total mineral matter recovered in Sections 21 and 22 plus the excess ash (Section 23) shall be moistened with a few drops of alcohol, boiled with 100 ml. of water, and washed successively through No. 6 (3360-micron), and No. 100 (149-micron) sieves with sufficient hot water to remove the fine mineral matter. The mineral matter retained on each sieve shall be collected separately, dried at 105 to 110 C. (221 to 230 F.) for 30 min., and weighed. From these weights, the weight of mineral matter shall be calculated and reported as follows:

Passing Sieve	Retained on Sieve	Weight of Mineral Matter per 108 sq. ft.
No. 6 (3360-micron)	No. 100 (149-micron) ..	.....
No. 100 (149-micron)[a]	.....	.....

[a] By difference.

(*b*) To check the weight of mineral matter passing a No. 100 (149-micron) sieve, the total aqueous suspension of mineral matter which passes through the No. 100 sieve shall be recovered by filtering through a weighed Gooch crucible, dried at 105 to 110 C. (221 to 230 F.), and weighed.

Analysis of Heavy Butt Shingles

26. (*a*) Specimens 2 in. in width and 8 in. in length shall be cut from the thin portion of samples selected in accordance with Section 15. These specimens shall be analyzed in accordance with the procedures described in Sections 20 to 25, inclusive, except that the calculation of the weight of the weather side coating shall be made by subtracting from the original weight of the sample the combined weight of the felt, the saturant in the felt, and the mineral matter passing a No. 6 (3360-micron) sieve and retained on a No. 100 (149-micron) sieve. (It is assumed that all granular surfacing and the greater portion of talc surfacing will be retained on a No. 100 (149-micron) sieve, and that the mineral filler will pass through.)

(*b*) Specimens from the thick portion of the shingle shall be secured and analyzed in accordance with the procedure described in Paragraph (*a*).

(*c*) The composition of the shingle as a whole, with reference to the specified requirements, shall be calculated from the combined areas of the respective portions analyzed in Paragraphs (*a*) and (*b*).

NOTE.—The method of analysis described in this section is not suitable for analyzing thick butt shingles of the "Tapered Type."

Tentative Method of Test for

KEROSINE NUMBER OF ROOFING AND FLOORING FELT BY THE VACUUM METHOD[1]

A.S.T.M. Designation: D 727 – 43 T

ISSUED, 1943.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. This method of test for the absorptive qualities of felt, expressed as kerosine number, is applicable to felts used in the manufacture of roofing and flooring.

NOTE.—The kerosine number of a felt, as determined by this method, is based on the relation between the specific gravity of the kerosine used and the specific gravity of water (1.00). To obtain the relation between the saturating capacity of the felt and any bituminous saturant to be used with the felt, the specific gravity at 77 F. (25 C.) of the bitumen shall be determined in accordance with the Standard Method of Test for Specific Gravity of Road Oils, Road Tars, Asphalt Cements, and Soft Tar Pitches (A.S.T.M. Designation: D 70) of the American Society for Testing Materials,[3] and that figure multiplied by the "kerosine number" of the felt, and expressed as a percentage figure.

Kerosine Number

2. The kerosine number of roofing and flooring felt is calculated from the maximum weight of a kerosine oil, of known specific gravity, retained by the felt after displacement of all the air from the interior voids. It is a measure of the amount of saturant which a given felt will absorb.

Apparatus

3. The apparatus shall consist of the following:

(*a*) *Balance.*—Analytical balance, sensitive to 1 mg.

(*b*) *Oven.*—Laboratory oven, steam or electrically heated and capable of maintaining a temperature between 220 and 225 F., inside dimensions to be not less than 12 by 12 by 12 in.

(*c*) *Weighing Container.*—Lightweight glass or metal container with tight-fitting cover for weighing the specimens, and of a suitable size to hold the specimens in a horizontal position without bending or distorting them.

(*d*) *Thermo-Hydrometer.*—Glass hydrometer and thermometer combined, also a glass hydrometer cylinder as prescribed in the Standard Method of Test for Gravity of Petroleum and

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

[2] Accepted by the Society at annual meeting, June, 1943.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Petroleum Products by Means of the Hydrometer (A.S.T.M. Designation: D 287) of the American Society for Testing Materials.[4]

(*e*) *Vacuum Pump.*—A vacuum pump capable of reaching and maintaining a vacuum of not less than 28 in. of mercury in the test cylinder.

(*f*) *Vacuum Cylinder.*—A glass vessel of approximately 1500 cu. cm. capacity for soaking the felt specimens in kerosine under vacuum.

(*g*) *Desiccator.*—A desiccator of size suitable for cooling felt strips in the weighing containers.

Kerosine

4. The water-white kerosine used in the test shall have a specific gravity between 0.776 and 0.825 at 25 C. (77 F.).

Test Specimens

5. Six representative specimens each measuring 2 ± $\frac{1}{8}$ in. by 5 ± $\frac{1}{8}$ in. in size shall be selected from the felt to be tested. The specimens shall be cut with the 5-in. side running parallel to the machine direction of the sheet.

Procedure

6. (*a*) Place the test specimens in the tared weighing container and expose them, uncovered, for not less than 1 nor more than 2 hr. in the oven, the interior of which is maintained uniformly at a temperature between 220 and 225 F. The specimens shall be kept flat; they shall not be folded, rolled, or in any way distorted. Handle the six specimens as a unit in all of the following operations.

(*b*) Remove the specimens from the oven in the weighing container and place, still uncovered, in the desiccator to cool.

(*c*) Rapidly seal the weighing container with its cover and weigh the container and included specimens on the analytical balance to the nearest 10 mg. Calculate the net weight of the dry specimens.

(*d*) Insert a wire hook in one corner of the specimens and immerse them at once in a vertical position in 1 liter of kerosine at 25 ± 1 C. (77 ± 1.8 F.) in the glass vacuum vessel. Apply a vacuum of not less than 28 in. of mercury to the vessel containing the specimens and hold them under that condition for 15 min., or until the bubbles cease to come from the specimens, whichever period is the longer. Remove the specimens from the kerosine and permit them to drain in the machine direction of the felt fibers for 3 min. ± 1 sec., allowing the lower corner of each specimen to touch the edge of the kerosine container.

(*e*) Return the specimens to their weighing container, seal with its cover, and determine and record the combined weight of the specimens and the kerosine which they have absorbed.

(*f*) Determine the specific gravity at 25 C. (77 F.) of the kerosine used in the test in accordance with the Standard Method of Test for Gravity of Petroleum and Petroleum Products by Means of the Hydrometer (A.S.T.M. Designation: D 287) of the American Society for Testing Materials.[4]

Calculation and Report

7. (*a*) Calculate the kerosine number of the specimens as follows:

$$\text{Kerosine number} = \left(\frac{b}{a} - 1\right) \times \left(\frac{1}{g} \times 100\right)$$

where:

a = weight of dry felt,
b = weight of felt plus absorbed kerosine, and
g = specific gravity of kerosine at 25 C. (77 F.)

(*b*) *Report.*—Report the results of the kerosine number determinations to three significant figures.

[4] 1944 Book of A.S.T.M. Standards, Part III.

Tentative Recommended Practice for

ACCELERATED WEATHERING TEST OF BITUMINOUS MATERIALS[1]

A.S.T.M. Designation: D 529 - 39 T

ISSUED, 1939.[2]

This Tentative Recommended Practice of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. This recommended practice is intended to produce rapid deterioration of bituminous materials under conditions simulating extreme outdoor exposure.

Apparatus

2. The apparatus required consists of an enclosed carbon-arc lamp, a cylinder, sprinkler, spray, and an arrangement for temperature control during the light period. The general arrangement of the cylinder and lamp as shown in Fig. 1 is suitable for testing materials in a vertical position, such as the harder bituminous compounds and roofings. Soft materials, of necessity, should be tested in a horizontal position and a mercury-arc lamp is better adapted in that case than a carbon-arc lamp.

(*a*) *Carbon-arc Lamp.*—The carbon-arc lamp (Note) shall operate on a 220-v. line, alternating current. The voltage shall be reduced by means of a resistance to 130 to 145 v. The lamp shall operate at 15 to 17 amp., and shall have approximately the following spectral range:[3]

Spectral Range, μμ	Percentage Total Radiation to Limit of Fluorite Transmission in Infra Red (12,000 μμ) Alternating Current Arc, 60 cycles, 15 to 17 amp.
279 to 290	0.8
290 to 320	4.1
320 to 360	6.0
360 to 480	14.5
480 to 600	8.0
600 to 1 400	14.8
1400 to 4 200	21.4
4200 to 12 000	30.4

Under these conditions the intensity of the light (15 in. opposite the flame) shall be between 3500 to 4000 foot-candles.

NOTE.—The alternating carbon-arc lamp described in this method is taken as the standard as it is now in general use. Mercury-arc, direct-current carbon-arc lamps, and other types of alternating-current carbon-arc lamps if employed must be defined in relation to the standard lamp.

(*b*) *Cylinder.*—An open, metal cylinder 30 in. in diameter and about 15 in. in depth, equipped with slots or hooks

[1] Under the standardization procedure of the Society, this recommended practice is under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] Accepted by the Society at annual meeting, June, 1939.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part p. 379 (1933).

to hold the test specimens and connected with a revolving mechanism, geared to furnish one revolution in 20 min., shall surround the lamp. The cylinder shall be equipped, in the lower central portion with a rotary sprinkler (Fig. 2) and with a spray placed vertically, close to the inner rim (Fig. 1). The lamp and cylinder shall be suspended over a pan (sump) about 4 to 5 in. in depth, the latter equipped with an overflow pipe.

(*c*) *Temperature Control.*—For maintaining the test specimens at a uniform temperature during the light period the weathering apparatus shall be equipped with some suitable means of automatic temperature control.

(*d*) *Refrigerator.*—A refrigerator capable of maintaining a temperature of −10 F. (−23 C.) may be used as a separate unit when a cold period is desired as a feature of the test cycle.

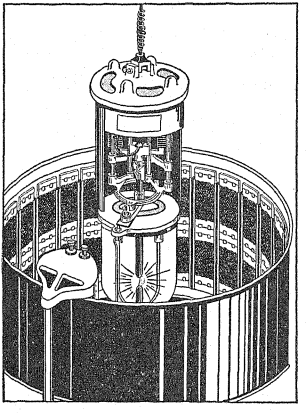


FIG. 1.—Apparatus for Accelerated Weathering Test.

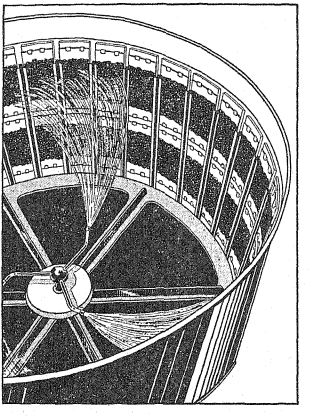


FIG. 2.—View of Apparatus Showing Rotary Sprinkler.

Test Specimens

3. The test specimens shall be approximately 6 in. in length and 3 in. in width. Bitumens and bituminous compounds shall be melted and spread to a uniform thickness over one surface of an aluminum plate of about $\frac{1}{16}$ in. in thickness. Fabricated materials, such as bituminous roofing materials, shall be cut to size and their weather surface exposed directly to the test cycle. If these are too flexible to sustain their own weight they may be mounted upon an aluminum plate.

Procedure

4. (*a*) The test specimens shall be subjected to a daily weathering cycle

consisting of periods of exposure to cold, light, and water. The weathering cycle (Note) shall be one of the other of the following three types according to the purpose of the test.

NOTE.—Three cycles are offered because experience has taught that weather varies considerably from place to place. The difference between the three cycles is principally in the water treatment; cycle A is a fairly "dry" cycle, cycle B is wetter, while cycle C is the wettest and simulates a hot humid climate. Any of these cycles will determine the effect of the weathering upon bituminous materials.

	For a Period of
CYCLE A[4,a]	
Cold exposure	1¾ hr.
Water exposure	1 hr.
Light exposure	1½ hr.
Water exposure	2 hr.
Light exposure	16½ hr.
CYCLE B[5]	
Water exposure	½ hr.
Cold exposure	1¾ hr.
Light – spray	4 hr.
Water exposure	½ hr.
Light exposure	17 hr.
CYCLE C[6]	
Light – spray	22 hr.
Cold exposure	1¾ hr.

[a] To change from one exposure period to the other requires approximately 15 min.

(*b*) For the light, rain, and light-spray exposure periods, the specimens shall be placed in the holders of the cylinder. For the rain period the lamp shall be raised above the cylinder, the latter covered, and the specimens sprinkled vigorously with water by means of a rotary spray which operates at the bottom of the cylinder.

(*c*) For the cold exposure period, the specimens shall be placed in the refrigerator, previously cooled to −10 F. (−23 C.). To change the panels from the cylinder to the refrigerator shall not require more than 15 min. and the same time may again be allowed after the cold period to return the specimens to the cylinder for the light-spray period.

(*d*) For the light period, the lamp shall be operated with the revolving cylinder. During the light-spray period, the light from the lamp shall shine on the specimens continuously while the water, running constantly from the spray at a fixed point, shall wet the specimens in the revolving cylinder once every 20 min.

(*e*) The air temperature during the light period shall be 140 ± 5 F. (60 ± 2.8 C.) This temperature shall be measured by means of a transparent-bulb, mercury thermometer placed behind the aluminum panel holder so that the bulb of the thermometer is shielded from the arc light by the panel holder between the two tiers of test specimens and shall be half way between the panel holder and the cylinder of the weathering apparatus.

(*f*) The radiation temperature during the light period shall be 180 ± 5 F. (82 ± 2.8 C.) This temperature shall be measured by means of a mercury thermometer, the bulb of which is thinly coated with unweathered asphalt. This thermometer shall be mounted inside the rotating cylinder at a distance of ¼ in. from the surface of the center of the panels and with its bulb on a level with the center of the panel holder between the two tiers of test specimens.

(*g*) In the case of the light-spray period, the specimens will, of course, be cooled as they pass by the water spray; the specimens should, however, warm to about these standard temperatures before being wet again. (At times it may be necessary to use warm water for spraying in order to reach such a

[4] This cycle is described in *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 381 (1933).

[5] This cycle is described in *Proceedings*, Am. Soc. Testing Mats., Vol. 37, Part I, p. 419 (1937).

[6] This cycle, without the cold exposure period, is described in a paper by Hubert R. Snoke and Braxton E. Gallup on "Accelerated Weathering Tests of Mineral-Surfaced Asphalt Shingles," *Journal of Research*, Nat. Bureau Standards, Vol. 18, No. 6, p. 669 (1937) *Research Paper 1002*.

temperature). Water (2 to 4 in. in depth) shall be kept in the tank or sump all the time, the excess water flowing out over the overflow.

(*h*) The position of the test specimens in the revolving cylinder shall be changed for each repetition of any one of the cycles, especially with respect to their location in the upper or lower tiers of slots or hooks, in order to compensate for unavoidable differences in their relation to the light source.

Determining Extent of Weathering

5. The result of the accelerated weathering tests shall be determined by comparing specimens of the unweathered material with test specimens that have been weathered. The durability of the material may be expressed as the number of cycles of weathering that are required to bring about definite changes in the pliability, breaking strength, solubility in standard solvents, and appearance of the material tested.

Tentative Methods of

TESTING VENEER, PLYWOOD, AND OTHER WOOD AND WOOD-BASE MATERIALS[1]

A.S.T.M. Designation: D 805 – 44 T

ISSUED, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These methods cover procedures for determining the following properties of veneer, plywood, and other wood and wood-base materials:

(*a*) Compression:

(*1*) Load applied parallel or perpendicular to the grain direction for material over $\frac{1}{4}$ in. in thickness,

(*2*) Load applied parallel or perpendicular to the grain direction for material $\frac{1}{4}$ in. in thickness or less, and

(*3*) Load applied at an angle to the grain direction,

(*b*) Static bending,

(*c*) Tension,

(*d*) Panel shear, Forest Products Laboratory Test (FPL),

(*e*) Plate shear, Forest Products Laboratory Test (FPL),

(*f*) Toughness,

(*g*) Hardness, and

(*h*) Moisture content and specific gravity.

NOTE.—The expanding use of plywood, wood, and wood-base laminated materials has stimulated an interest in the mechanical properties of these materials, and in methods for evaluating their mechanical properties. In the preparation of these methods of test, special consideration has been given to procedures already in satisfactory use in the determination of the mechanical and physical properties. Consideration has been given also to the correlation of the methods with related procedures already in use or under consideration for other materials.

The mechanical tests described in these methods may be made to obtain strength data for design purposes, to determine the effect on strength of various treatments or of various factors in processing, to ascertain properties in relation to the various grain or fiber directions of the material, to compare the properties of different base materials or different species, and for other similar purposes.

Test Specimen

2. (*a*) *Selection of Test Specimens.*—The number of test specimens to be chosen, and the method of their selection depend on the purpose of the particular tests under consideration, and no general rule can be given to cover all cases. However, if specimens are to be used for acceptance tests, not less than five speci-

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-7 on Wood.

[2] Accepted by Committee E-10 on Standards, August 28 1944.

mens of a type shall be tested. Specimens shall not be taken closer to the edge of the sheet or panel than twice the thickness of the sheet or panel.

(*b*) *Control of Moisture Content.*—All test specimens shall be brought to constant weight before testing, preferably in a conditioning room with temperature and humidity control. Because of the influence of moisture and temperature on the mechanical properties, the tests shall preferably be made in a room under temperature and humidity control. A temperature of 72 F. and a relative humidity of 63 per cent is recommended, which corresponds in wood to a moisture content of about 12 per cent, based on the oven-dry weight. In any event, the tests shall be conducted so that the moisture content will be uniform throughout the thickness, and so that changes in the moisture content of the specimens will not occur during the test.

Report

3. The report of each test shall include a complete description of the material. For plywood, the report shall include the species and thickness of each ply, the grain direction, the type of glue, the kind of veneer with respect to method of cutting, the pressing temperature, and the pressure for each panel; for the wood and wood-base materials the report shall include the essential information on the base material, the method of treatment or impregnation, if any, the kind and amount of impregnating material used, the direction of the grain or fibers, the temperature and pressure used, and the thickness.

COMPRESSION PARALLEL OR PERPENDICULAR TO GRAIN

Direction of Grain or Fibers

4. The grain direction of the individual plies or laminations shall be parallel or perpendicular to the length of the test specimen.

Test Specimen

5. (*a*) The test specimen shall be rectangular in cross-section. The width and thickness shall be measured to the nearest 0.001 in. and the length to the nearest 0.01 in.

(*b*) *Material Over* $\frac{1}{4}$ *in. in Thickness.*—For material over $\frac{1}{4}$ in. in thickness, the specimen shall have a thickness equal to that of the material and the width shall be a minimum of 1 in. but not less than the thickness. The length shall be not greater than seven times the least cross-sectional dimension.

(*c*) *Material* $\frac{1}{4}$ *in. or Less in Thickness.*—For material $\frac{1}{4}$ in. and less in thickness, the specimen shall have a thickness equal to that of the material and the width shall be nominally 1 in. The length shall be 4 in. Such specimens shall be supported laterally throughout the test.

End Surfaces Parallel

6. Care shall be taken in preparing the test specimens to make the end surfaces smooth and parallel to each other and at right angles to the length.

Lateral Support

7. Test specimens $\frac{1}{4}$ in. or less in thickness shall be supported laterally to prevent buckling during the test, but undue pressure shall not be exerted against the sides of the specimen. This support shall not measurably restrain the normal compressive deformation under load. A satisfactory method of providing lateral support is illustrated in Fig. 1 and a detailed design of this apparatus is shown in Fig. 2.

Loading Procedure

8. The load shall be applied through a spherical bearing block preferably of the

suspended, self-aligning type. The load shall be applied with a continuous motion of the movable head to maximum load at a rate of 0.003 in. per in. of length of the specimen per minute within a permissible variation of plus or minus 25 per cent.

NOTE.—After the maximum load has been reached, the rate of loading may be increased to develop failure.

from the specimen ends. Figure 3 shows a type of *Lamb's* roller compressometer, with a 2-in. gage length that has been found satisfactory for specimens $3\frac{1}{2}$ to 6 in. in length. A suitable arrangement of a Marten's mirror having a 2-in. gage length for measuring deformations of laterally supported specimens is illustrated in Fig. 1.

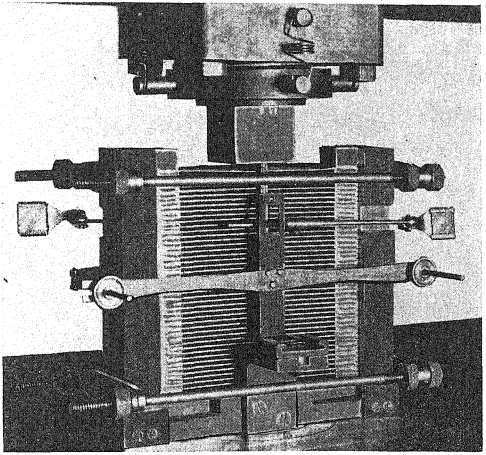


FIG. 1.—Compression Test of Thin Plywood Supported Laterally Showing Marten's Mirror Compressometer for Measuring Deformations on Two Opposite Faces.

Load-Deformation Curves

9. Data for load-deformation curves may be taken to determine the modulus of elasticity and the proportional limit. Increments of load shall be chosen so that not less than 12 and preferably 15 or more readings of load and deformation are taken to the proportional limit. The deformation shall be read to the nearest 0.0001 in. Compressometers shall be attached over the central portion of the length of the specimen and the points of attachment shall be not less than $\frac{3}{4}$ in.

Moisture and Specific Gravity

10. The moisture content and specific gravity of each test specimen shall be determined as described in Sections 52 and 53.

COMPRESSION AT AN ANGLE TO THE GRAIN

Test Specimen

11. The specimen shall be rectangular in cross-section. The thickness of the specimen shall be equal to the thickness of the material (Note) and the height

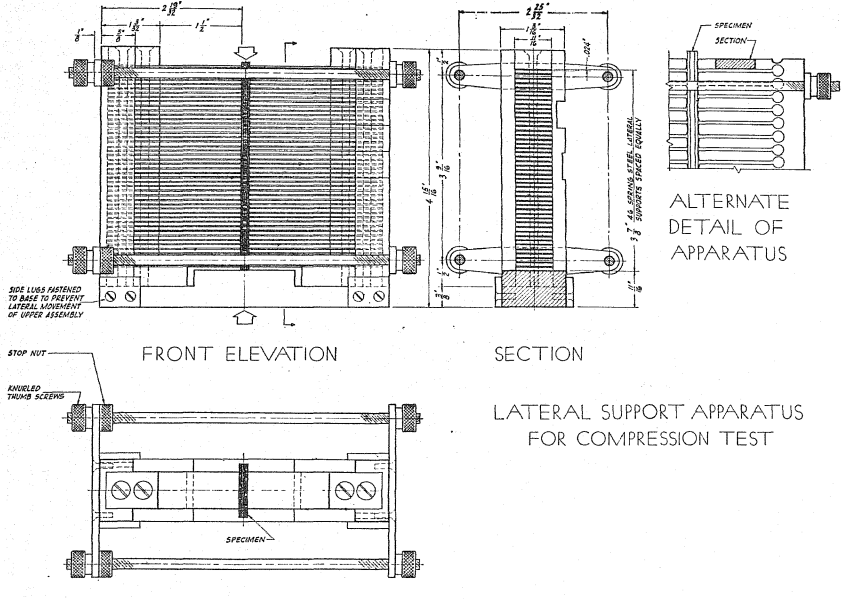


FIG. 2.—Apparatus for Lateral Support for Compression Test.

shall be not greater than seven times the thickness. The width shall be not less than the height nor more than four times the height. The thickness shall be measured to the nearest 0.001 in. and the width and height to the nearest 0.01 in.

NOTE.—This method of test is applicable only to material $\frac{1}{4}$ in. or greater in thickness.

the suspended, self-aligning type. When the test specimen is of considerable width, it may be necessary to transmit the load to the specimen by means of a rigid, rectangular steel bar. The load shall be applied with a continuous motion of the movable head to maximum load at a rate of 0.003 in. per in. of length of the

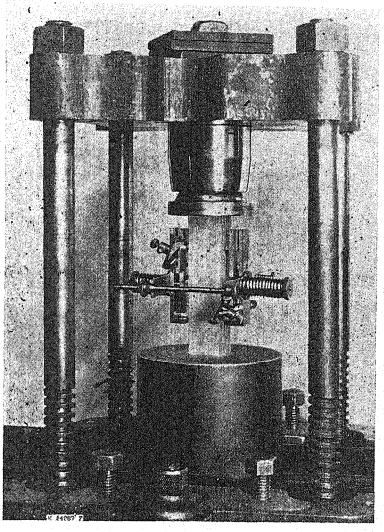


FIG. 3.—Compression Test of Plywood Prism Showing Roller Compressometer Attachment (2-in. Gage Length) Affording Average Deformation for Two Opposite Faces.

End Surfaces Parallel

12. Care shall be taken in preparing the test specimens to make the end surfaces smooth and parallel to each other and at right angles to the height.

Loading Procedure

13. The load shall be applied through a spherical bearing block preferably of specimen per minute within a permissible variation of plus or minus 25 per cent.

Load-Deformation Curves

14. Data for load-deformation curves may be taken to determine the modulus of elasticity and the proportional limit. Increments of load shall be chosen so that not less than 12 and preferably 15 or more readings of load and deformation

are taken to the proportional limit. The deformation shall be read to the nearest 0.0001 in. Compressometers shall be attached over the central portion of the length of the specimen and the points of attachment shall be not less than $\frac{3}{4}$ in. from the specimen ends. Figure 4 shows a Marten's mirror compressometer

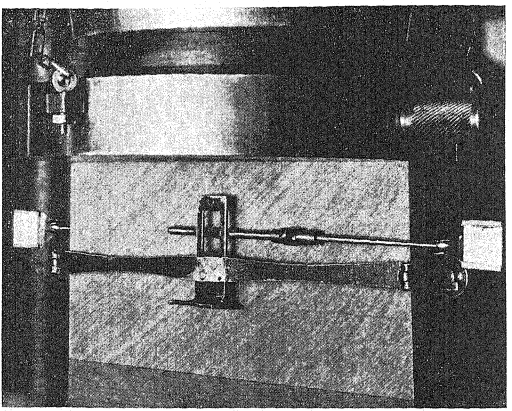


FIG. 4.—Compression Test of Plywood Showing Type of Specimen Used When Load is Applied at an Angle Other Than 0 or 90 deg. to the Direction of Grain and Application of Marten's Mirror-Type Compressometer.

with a 1-in. gage length that has been found satisfactory.

Moisture and Specific Gravity

15. The moisture content and specific gravity of each test specimen shall be determined as described in Sections 52 and 53.

STATIC BENDING

Direction of Grain or Fibers

16. The grain direction of the individual plies or laminations shall be parallel or perpendicular to the span length.

Test Specimen

17. The test specimen shall be rectangular in cross-section. The depth of the specimen shall be equal to the thickness of material and the width shall be 1 in. for depths not exceeding 1 in., and 2 in. for greater depths. When the grain direction of the face plies or laminations is parallel to the span, the length of the specimen shall be not less than 48 times the depth plus 2 in.; when the grain direction of the face plies or laminations is perpendicular to the span, the specimen length shall be not less than 24 times the depth plus 2 in. The actual width and depth shall be measured to the nearest 0.001 in. and the specimen and span length shall be measured to the nearest 0.01 in.

Span and Supports

18. Center loading shall be used. The

span shall be not less than 48 times the depth when the grain direction of the face plies or laminations of the test specimen is parallel to the span and not less than 24 times the depth when the grain direction of the face plies or laminations is perpendicular to the span. A 1-in. overhang shall be allowed at each end (see Section 17). Round-nosed knife-edges or roller-bearing supports may be used. In both instances, the supports shall be adjustable laterally to compensate for slight twist or warp in the specimen. The construction of this apparatus is shown in detail in Fig. 5. In cases where excessive local deformation may occur, suitable bearing plates shall be used.

Loading Block

19. A loading block having a radius of curvature of one and one-half times the depth of the test specimen for a chord length of not less than twice the depth of the specimen shall be used. In cases where excessive local deformation may occur, suitable bearing plates shall be used.

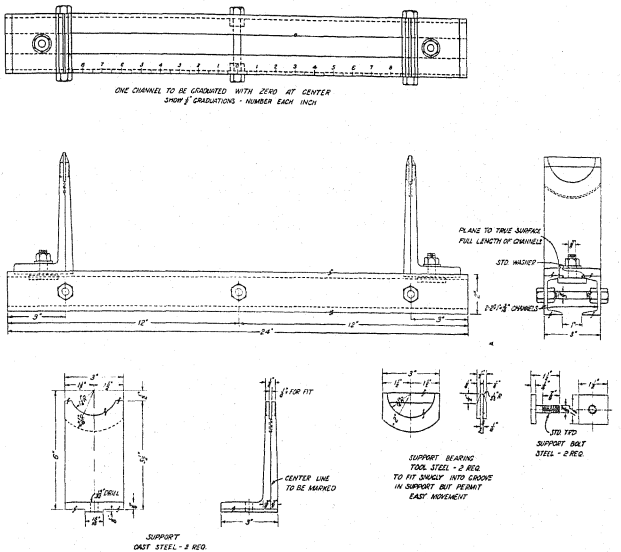


FIG. 5.—Apparatus for Static Bending Test Showing Details of Laterally Adjustable Supports.

Loading Procedure

20. The load shall be applied with a continuous motion of the movable head throughout the test. The rate of load application shall be such that the unit rate of fiber strain is equal to 0.0015 in. per in. of outer fiber length per minute

within a permissible variation of plus or minus 25 per cent. The rate of motion of the movable head may be calculated as follows:

$$N = \frac{zL^2}{6d}$$

proportional limit, work to maximum load, and total work. Deflections may be taken by the methods indicated in Figs. 6 or 7, and readings shall be taken to the nearest 0.001 in. Increments of load shall be chosen so that not less than

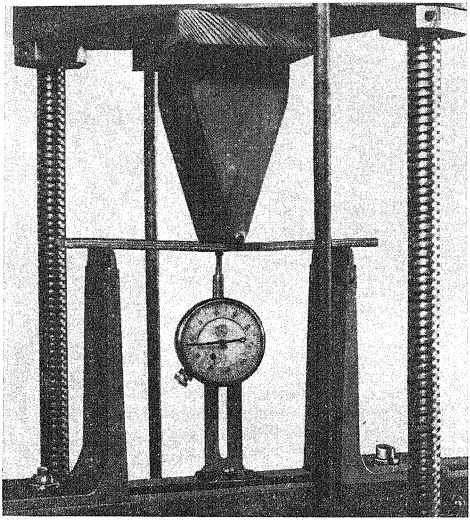


FIG. 6.—Static Bending Test of Plywood Showing Adjustable Supports and One Method of Attaching Dial Gage for Observing Deflections of Thin Material.

where:

N = rate of motion of moving head, in inches per minute,
L = span, in inches,
d = depth of beam, in inches, and
z = unit rate of fiber strain, in inches per inch of outer fiber length per minute (0.0015).

Load-Deflection Curves

21. Data for load-deflection curves may be taken to determine the modulus of elasticity, proportional limit, work to 12 and preferably 15 or more readings of load and deflection are taken to the proportional limit.

Moisture and Specific Gravity

22. The moisture content and specific gravity of each test specimen shall be determined as described in Sections 52 and 53.

TENSION

Test Specimen

23. The size and shape of the test

specimen shall be selected from the three types (A, B, or C) shown in Fig. 8. The basis for selection shall be the grain angle and the thickness of the material. If the grain of the individual plies or laminations makes an angle other than 0 or 90 deg. with the length of the specimen, type C shall be used regardless of the thickness of the material. Type A shall be used for material over $\frac{1}{4}$ in. in thickness and type B for material $\frac{1}{4}$ in. or less in thickness. The specimen shall have a thickness equal to that of the material and it shall be measured to the nearest 0.001 in.; the width at the critical section shall also be measured to the nearest 0.001 in.

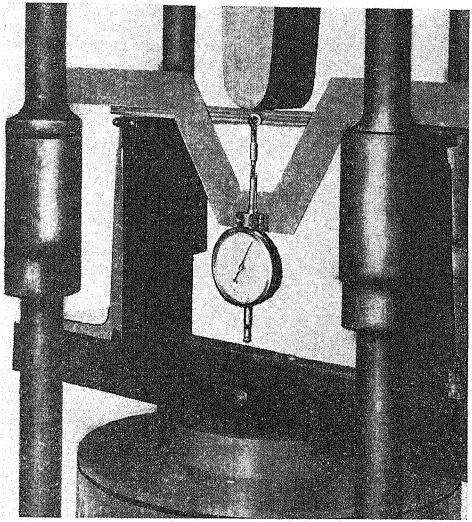


FIG. 7.—Static Bending Test on Thick Plywood Showing Roller Bearing at Supports and Special Yoke with Dial Gage for Measuring Deflection at the Neutral Axis.

Shaping of Specimen

24. The test specimens shall be properly shaped using a template in conjunction with a vertical-spindle woodworking shaper or any other method that will give equally satisfactory results.

Loading Procedure

25. The load shall be applied continuously throughout the test at a rate of motion of the movable head of 0.035 in. per min. The specimen shall be held in wedge-type self-tightening and self-aligning grips.

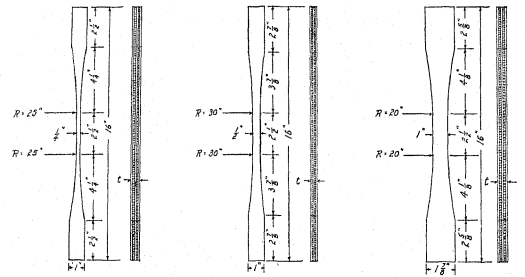
Load-Deformation Curves

26. Data for load-deformation curves may be taken to determine the modulus of elasticity and the proportional limit. Increments of load shall be chosen so that not less than 12 and preferably 15 or more readings of load and deformation

are taken to the proportional limit. The deformation apparatus shall be attached at the central portion of the length of the specimen at the center of the portion of constant cross-section. Deformation readings shall be taken to the nearest 0.0001 in. Figures 9 and 10 show 2-in. gage length Tripolitis extensometers that have been found satisfactory for this test.

Moisture and Specific Gravity

27. The moisture content and specific gravity of each test specimen shall be determined as described in Sections 52 and 53. For determining specific gravity, a separate specimen closely matched to the tension specimens shall be used.



NOTE.—A generous radius of curvature at the minimum section as provided in this specimen is highly desirable

FIG. 8.—Dimensions and Details of Plywood Tension Test Specimens.

PANEL SHEAR (FOREST PRODUCTS LABORATORY TEST)

Direction of Grain

28. The grain direction of the individual plies shall be at an angle of 0 or 90 deg. with the edges of the panel.

Test Specimen

29. The dimensions of the test specimen (Figs. 11 and 12) shall conform to the sizes prescribed in Table I for the respective thickness of plywood used. The size and thickness of reinforcing blocks (hard maple or birch) shall also conform to the values prescribed in this table. The thickness of the specimen shall be measured to the nearest 0.001 in. and the actual width and length of the panel shall be measured to the nearest 0.01 in.

Size and Location of Pins

30. The sizes and location of the pins used to load the panel shall be as prescribed in Table I. Care shall be taken in drilling the holes to make the surfaces smooth and the axes perpendicular to the plane of the plywood.

Pins and Rollers

31. The pins shall be of a quality equal to that of steel having a yield point of approximately 100,000 psi. and an ultimate tensile strength of approximately 125,000 psi. The rollers shall be the ball or roller-bearing type of standard bearing sizes with an inside diameter approximately 0.001 in. larger than the pin diameter. Where necessary a sleeve bushing shall be pressed into the bearing to achieve this tolerance. For pin diameters in excess of 0.312 in., double-row ball bearings or roller bearings of

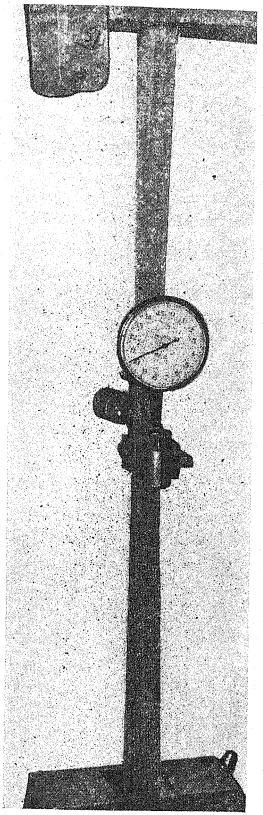


FIG. 9.—Tension Test of Thick Plywood Showing Nonaveraging Type of Extensometer (2-in. Gage Length).

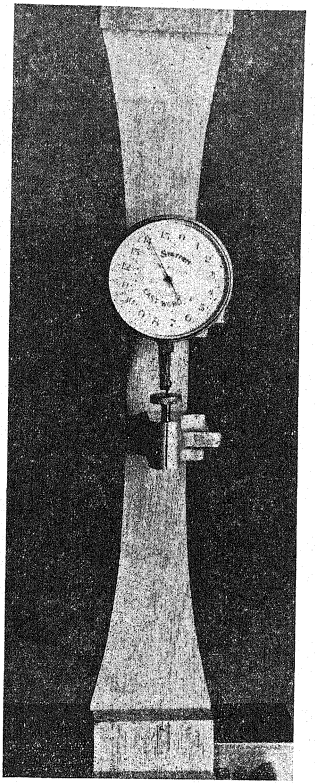


FIG. 10.—Tension Test of Plywood with Extensometer Attachment Showing Specimen Used When Load is Applied at an Angle Other Than 0 or 90 deg. to the Direction of the Grain of the Plies.

equivalent strength shall be used. The outside bearing diameter shall be equal to that prescribed in Table I.

Loading Procedure

32. The load shall be applied by special steel loading blocks which articulate with

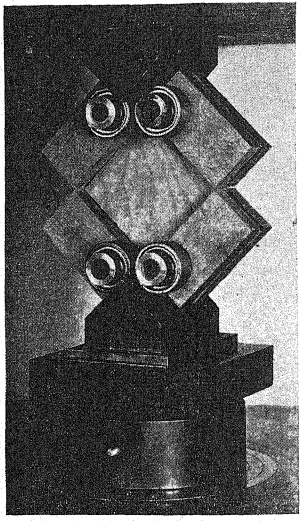


FIG. 11.—Panel Shear Test (F.P.L.) Showing Specimen Used and Method of Loading. Strain gages may be attached if necessary or desired.

the rollers and pins attached to the test specimen. The angle between faces of the loading block shall be 90 deg. and between each face and the base the angle shall be 45 deg. A spherical bearing block, preferably of the suspended, self-aligning type shall be employed in the loading system. The load shall be applied continuously throughout the test with a uniform motion of the movable head of the testing machine equal to 0.005 in. per in. of length of diagonal per minute within a permissible variation of plus or minus 25 per cent.

Load-Deformation Curves

33. Data for load-deformation curves may be taken to determine the modulus of elasticity and the proportional limit. Strain gages shall be attached to the central portion of a diagonal on opposite sides of the specimen. The deformation shall be read to the nearest 0.0001 in.

Calculation

34. The unit shear stress on the plywood panel shall be calculated as follows:

$$f_s = \frac{0.707\ P}{Lt}$$

where:

f_s = unit shear stress, in pounds per square inch,
P = total applied load, in pounds,
L = side of square panel specimen (see Fig. 12), and
t = thickness of specimen (see Fig. 12), in inches.

Moisture and Specific Gravity

35. After the test a section shall be cut out of the panel, weighed and measured, and used to determine the moisture content and specific gravity of the specimen as described in Sections 52 and 53.

PLATE SHEAR (FOREST PRODUCTS LABORATORY TEST)

Direction of Grain

36. The grain direction of the individual plies or laminations shall be parallel or perpendicular to the edges of the test specimen.

Test Specimen

37. The test specimen shall be square with the thickness equal to the thickness of the material and the length and width not less than 25 nor more than 40 times the thickness. The thickness shall be

TABLE I.—DIMENSIONS OF PLYWOOD PANEL SHEAR SPECIMENS.
(To be used with Fig. 12)

Plywood Thickness *t*, in.	Maximum Allowable Panel Size (between blocks), *L*, in.	Minimum Block Width, *w*, in.	Minimum Block Thickness, *t'*, in.	Diameter of Pins, *d*, in.	Diameter of Pin Hole, in.	Diameter of Rollers, *D*, in.	Inner Edge of Block to Center Line of Pin Hole, *a*, in.	End of Block to Center Line of Pin Hole, *b*, in.
GROUP I WOODS (HIGH-DENSITY SPECIES)								
0.05 to 0.10	1.00	1.00	1/4	0.250	1/4	0.8661	1/4	3/8
0.10 to 0.20	2.00	1.00	3/8	0.390	25/64	1.1811	5/16	1 5/32
0.20 to 0.30	3.00	1.50	3/4	0.587	19/32	1.3780	15/32	1 5/32
0.30 to 0.40	4.00	2.00	3/4	0.784	51/64	1.8504	5/8	1 1/16
0.40 to 0.50	5.00	2.50	1 5/16	0.981	63/64	2.4409	25/32	1 5/16
0.50 to 0.60	6.00	3.00	1 3/8	1.178	1 3/16	2.8346	15/16	1 7/16
GROUP II WOODS (MEDIUM-DENSITY SPECIES)								
0.05 to 0.10	1.00	1.00	3/16	0.250	1/4	0.8661	1/4	3/8
0.10 to 0.20	2.00	1.00	5/16	0.390	25/64	1.1811	5/16	1 5/32
0.20 to 0.30	3.00	1.25	5/8	0.520	17/32	1.3780	15/32	1 5/32
0.30 to 0.40	4.00	1.75	1 1/16	0.784	51/64	1.8504	5/8	1 1/16
0.40 to 0.50	5.00	2.25	7/8	0.875	57/64	2.4409	25/32	1 5/16
0.50 to 0.60	6.00	2.50	1	1.178	1 3/16	2.8346	15/16	1 7/16
GROUP III WOODS (LOW-DENSITY SPECIES)								
0.05 to 0.10	1.00	0.75	3/16	0.250	1/4	0.8661	1/4	3/8
0.10 to 0.20	2.00	1.00	5/16	0.312	5/16	1.0236	5/16	1 5/32
0.20 to 0.30	3.00	1.25	7/16	0.438	7/16	1.2598	13/32	1 5/32
0.30 to 0.40	4.00	1.50	9/16	0.587	19/32	1.3780	5/8	1 1/16
0.40 to 0.50	5.00	2.00	11/16	0.784	51/64	1.8504	25/32	1 5/16
0.50 to 0.60	6.00	2.50	7/8	0.875	57/64	2.4409	15/16	1 7/16

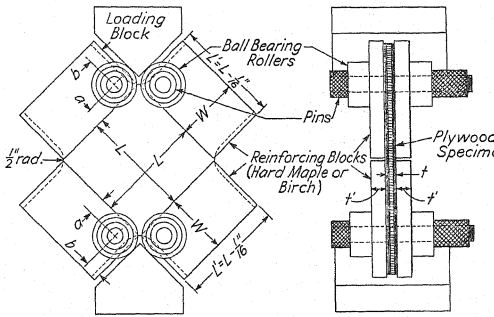


FIG. 12.—Plywood Panel Shear (F.P.L.) Frame Showing Size and Construction Details and Location of Pin and Rollers (To be used with Table I).

measured to the nearest 0.001 in. and the length and width to the nearest 0.01 in. Care shall be taken to avoid obtaining test specimens with initial curvature.

Loading Procedure

38. The test specimen shall be supported on rounded supports having a radius of curvature not greater than 1/4 in. on the opposite ends of a plate diagonal and loaded in a similar manner on the opposite ends of the other diagonal. In order that the loads may be applied at

the corners, metal plates shall first be attached as shown in Fig. 13. The loading and supporting frame shall be rigid. Figures 14 and 15 indicate the method of test and show details of the plate shear apparatus. The load shall be applied with a continuous and uniform motion of the movable head at a rate of 0.003 times the length of the plate in inches, expressed in inches per minute, within a permissible variation of plus or minus 25 per cent.

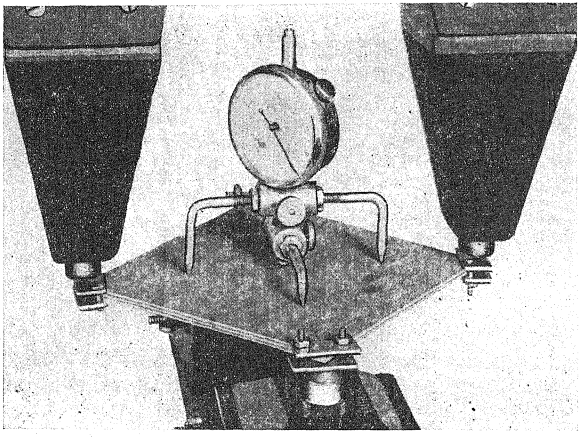


FIG. 13. Plate Shear Test (F.P.L.) of Plywood Showing Method of Loading and Observing the Differential Deformation Along the Two Diagonals.

Deformation Measurements

39. The deformation shall be measured to the nearest 0.001 in. at two points on each diagonal equidistant from the center of the plate. These measurements shall preferably be made at the quarter points of the diagonals and if other points than these are chosen, care shall be taken to avoid locations near the plate corners to avoid the load and reaction effects. The plate shall not be stressed beyond its elastic range and increments of load shall be chosen so that not less than 12 and preferably 15 load-deformation readings are taken. To eliminate the effects of slight initial curvature, two sets of data shall be obtained, the second set with the panel rotated 90 deg. about an axis through the center of the plate and perpendicular to the plane of the plies. The two results shall be averaged to obtain the shear modulus for the plate. A satisfactory arrangement for measuring relative deformations is indicated in Fig. 13; the dial readings in this case give twice the average deflection of the four points.

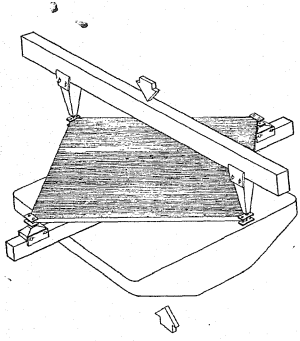


FIG. 14.—Method of Making Plate Shear Test.

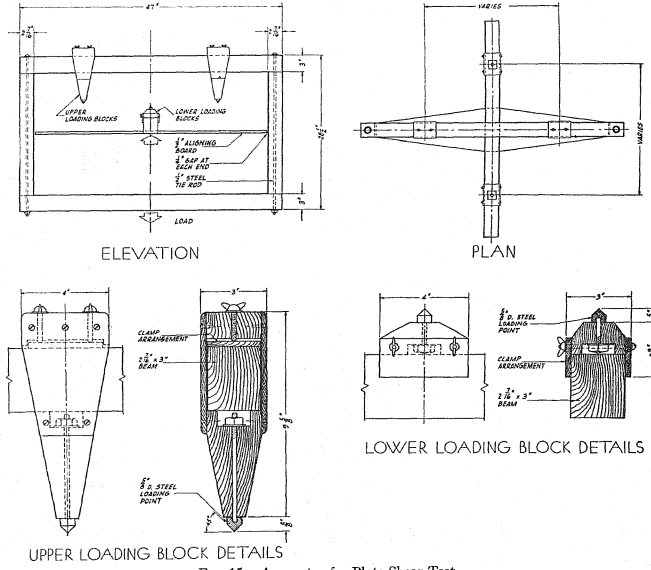


FIG. 15.—Apparatus for Plate Shear Test.

Calculation

40. The shearing modulus of elasticity shall be calculated as follows:

$$G = \frac{3u^2 P}{2h^3 w}$$

where:

G = shearing modulus, in pounds per square inch,
P = load applied to each corner, in pounds,
h = thickness of the plate, in inches,
w = deflection relative to the center, in inches, and
u = distance from the center of the panel to the point where the deflection is measured, in inches.

NOTE.—The average values of P and w are generally taken from the slope of a previously plotted load-deflection curve.

Moisture and Specific Gravity

41. The moisture content and specific gravity of each test specimen shall be determined as described in Sections 52 and 53. Where additional tests are to be made on the plate a satisfactory moisture specimen may be provided when the specimen is cut out, in which case this moisture specimen shall be subjected to the same conditioning as the plate proper.

NOTE.—This method of test is primarily designed for material in which the grain of the individual plies or laminations is parallel or perpendicular to the edge of the plate. It may be used, however, for plates in which the grain is at 45 deg. to the plate edges if a four-ply panel with all plies of the same thickness is used. The controlling condition is that the EI (modulus of elasticity × moment of inertia) values along both diagonals shall be equal.

TOUGHNESS

Test Specimen

42. The toughness test specimen to be used in conjunction with the Forest Products Laboratory toughness testing machine shall be $\frac{5}{8}$ in. in height for material less than $\frac{5}{8}$ in. in thickness and the thickness of the specimen shall be equal to that of the material. For material more than $\frac{5}{8}$ in. in thickness the height shall be equal to the depth and the depth shall be equal to the thickness of the material. The length shall be determined from the thickness of the material in accordance with Table II. The actual height and depth shall be measured to the nearest 0.001 in. and the specimen and span length shall be measured to the nearest 0.01 in.

TABLE II.—LENGTH OF TOUGHNESS TEST SPECIMENS.

Plywood Thickness, in.	Span Length, in.	Specimen Length, in.
Up to $\frac{1}{8}$, incl.	2	4
Over $\frac{1}{8}$ to $\frac{1}{4}$, incl.	3	5
Over $\frac{1}{4}$ to $\frac{3}{8}$, incl.	5	7
Over $\frac{3}{8}$ to $\frac{1}{2}$, incl.	6	8
Over $\frac{1}{2}$ to $\frac{5}{8}$, incl.	8	10
Over $\frac{5}{8}$ to $\frac{3}{4}$, incl.	9	11
Over $\frac{3}{4}$ to 1, incl.	12	14

Apparatus

43. The toughness testing machine (Note) shall consist of a frame supporting a pendulum, being so arranged that a measured amount of the energy from the fall of the pendulum may be applied to a test specimen. The pendulum shall consist of a bar to which is fastened a weight adjustable to different positions and shall carry at its upper end a drum or pulley whose center provides the axis of rotation. The force shall be applied to the specimen by means of a flexible steel cable passing over the drum. A stationary graduated scale or dial and a vernier operated by the moving drum shall be provided for reading the angles through which the pendulum swings. The machine shall be adjusted before test so that the pendulum hangs truly vertical and adjusted to correct for friction. The cable shall be adjusted so that the load is applied to the specimen when the pendulum swings to within approximately 15 deg. of the vertical so as to produce

complete failure by the time the downward swing is completed.

NOTE.—The set-up shown in Fig. 16 has been found to give satisfactory results.

Span and Supports

44. The test specimen shall be supported as a beam on two vertical pins, which exert reactions that are perpendicular to the plane of the plies or laminations. These pins shall be adjusted to the span length taken from Table I which provides for a specimen overhang of 1 in. at each end.

Loading Procedure

45. The load shall be applied at the center of the span perpendicular to the plane of the plies. The load shall be applied through a tup, attached to a flexible cable, having a radius of curvature approximately equal to one and one-half times the depth of the test specimen. The weight position and initial angle of the pendulum shall be chosen so that complete failure of the specimen is obtained on one drop. Most satisfactory results are obtained when the difference between the initial and final angle is at least 10 deg.

Calculation

46. The initial and final angle shall be read to the nearest 0.2 deg. by means of the attached vernier. The toughness shall then be calculated as follows:

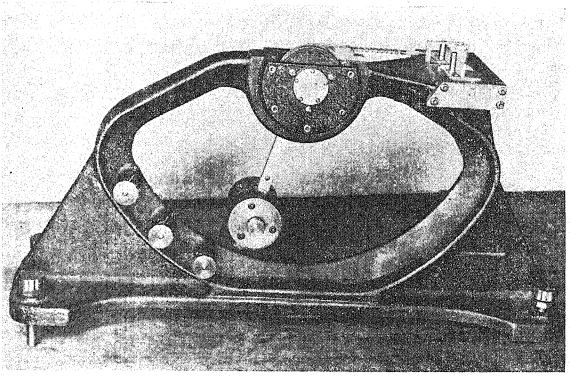


FIG. 16.—Toughness Test of Plywood Showing Load Applied to Specimen When Pendulum is Approximately 15 deg. from the Vertical.

$$T = wL(\cos A_2 - \cos A_1)$$

where:

T = toughness (work per specimen), in inch pounds,
w = weight of the pendulum, in pounds,
L = distance from the center of the supporting axis to the center of gravity of the pendulum, in inches,
A_1 = initial angle (Note), in degrees, and
A_2 = final angle the pendulum makes with the vertical after failure of the test specimen, in degrees.

NOTE.—Since friction is compensated for in the machine adjustment, the initial angle may be regarded as exactly 30, 45, or 60 deg., as the case may be.

Moisture and Specific Gravity

47. The moisture content and specific gravity of each test specimen shall be determined as described in Sections 52 and 53.

ROCKWELL HARDNESS

(For material with specific gravity equal to or greater than 1.0)

Test Specimen

48. The test specimen shall be at least 1 in. square, with a minimum thickness of 0.25 in. unless it has been verified that for the thickness used the hardness values are not affected by the supporting surface and that no imprint shows on the under surface of the specimen after testing. The surfaces of the specimen shall be smooth and normal to the axis of the penetrator and the specimen shall rest solidly on the anvil.

Apparatus

49. The standard Rockwell hardness tester described in the Standard Method of Test for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials (A.S.T.M. designation: E 18),[3] shall be used and the M scale employed, unless otherwise specified. The Rockwell M scale hardness test requires the use of a $\frac{1}{4}$-in. hardened steel ball penetrator, a minor load of 10 kg. and a major load of 100 kg., with readings taken on the red dial.

Procedure

50. (*a*) Without the test specimen in place the Rockwell tester shall be adjusted by means of the dash pot so that the time of application of the major load is from 4 to 5 sec. with the 100-kg. load. With the specimen in place, the minor load of 10 kg. shall be applied and the zero setting (130 on the red scale) made 10 sec. thereafter. The major load shall then be applied by tripping the crank handle. The major load shall be removed within 7 to 8 sec. thereafter and the hardness reading shall be made 45 sec. thereafter unless the pointer comes to rest in a shorter time.

(*b*) The Rockwell M hardness number may be more than 100 or may be negative. Determine the number of times the needle passes the zero on the red scale on application of the major load and subtract the number of times the needle passes zero on removal of the major load. If the result is zero the reading is over 100; if 1, 0 to 100; if 2, the reading is negative. The Rockwell hardness number and scale letter shall be given in reporting the hardness of the material.

Moisture Content and Specific Gravity

51. The moisture content and specific gravity of each test specimen shall be determined as described in Sections 52 and 53.

MOISTURE CONTENT AND SPECIFIC GRAVITY

Procedure

52. The test specimen shall be accurately weighed and measured at the test (Note 1). After the test a sample which is to be used to determine the moisture content, shall be cut from the body of the specimen (Note 2) near the point of failure. This moisture sample shall be weighed immediately and dried in an oven at 100 C. until approximately constant weight is attained. After drying, the sample shall be weighed immediately. The weights of the test specimens and of the moisture samples shall be determined to an accuracy of not less than 0.2 per cent.

NOTE 1.—Veneer, plywood, and laminated materials may splinter somewhat in certain

[3] 1942 Book of A.S.T.M. Standards, Part I.

tests, so that the original specimen may not be intact at the end of a test. In such instances, the specimens should obviously be weighed before test.

NOTE 2.—For specimens of thin material or of small size, it may be desirable to use the entire specimen as a moisture sample. In specimens where the original weight cannot be obtained (for example, the panel shear specimen) the sample must be treated as a new specimen and shall be measured as well as weighed prior to drying.

Calculations

53. (*a*) *Moisture Content.*—The moisture content (Note 1) shall be calculated as follows:

$$M = \frac{W - F}{F} \times 100$$

where:

M = moisture content, in per cent,
W = initial weight, and
F = final weight.

(*b*) *Specific Gravity.*—The specific gravity (Notes 1 and 2) shall be calculated as follows:

$$\text{Sp. gr.} = \frac{W \times 0.061}{\left(1 + \frac{m}{100}\right) \times L \times w \times t}$$

where:

W = original weight of the specimen, in grams,
m = moisture content of specimen at time of test, in per cent,
L = length of specimen, in inches,
w = width of specimen, in inches, and
t = thickness of specimen, in inches.

NOTE 1.—The moisture content and specific gravity as determined above are the average values for the entire specimen. In plywood made up of thin veneers, the glue may constitute a significant part of the total weight and as a result the calculated specific gravities and moisture contents may vary substantially from the true values for the veneers. In some instances it may be desirable to take this into account.

NOTE 2.—The specific gravity so obtained is based on the volume at test and weight when oven-dry. If desired, the specific gravity may be obtained on an oven-dry weight and volume basis, using the procedure described in the Standard Methods of Testing Small Clear Specimens of Timber (A.S.T.M. Designation: D 143).[4] In each instance, the basis of the specific gravity value with respect to volume and moisture condition shall be stated.

[4] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Tentative Specifications for

BLACK SYNTHETIC IRON OXIDE[1]

A.S.T.M. Designation: D 769 – 44 T

ISSUED, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These specifications cover the pigment commercially known as black synthetic iron oxide.

Composition and Properties

2. (*a*) The pigment shall be a manufactured ferro-ferric oxide obtained by chemical reaction. It shall be a soft dry finely disintegrated pigment, free of admixtures of other substances and shall conform to the following requirements:

Total ferrous and ferric oxide, min., per cent	96.0
Ferrous oxide FeO, min., per cent	20.0
Water soluble material, max., per cent	0.5
Moisture and other volatile matter, max., per cent	1.0
Coarse particles (total residue retained on a No. 325 (44-micron) sieve), max., per cent	0.5
Hydrogen ion concentration (pH value)	6.5 to 8.5

(*b*) The mass color and character of tint formed by mixture with a white pigment shall be the same as, and the strength not less than, that of a reference sample mutually agreed upon by the purchaser and the seller.

Number of Tests

3. Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10,000 lb., except that for shipments of less than 10,000 lb. two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

Methods of Testing

4. (*a*) *General.*—Tests shall be conducted in accordance with the appropriate methods of the American Society for Testing Materials, where applicable. Test procedures not covered by A.S.T.M. methods shall be mutually agreed upon by the purchaser and the seller.

(*b*) *Hydrogen Ion Concentration Test.*—Hydrogen ion concentration shall be

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Accepted by the Society at annual meeting, June, 1944.

determined in accordance with either of the following methods:

(*1*) *Electrometric Method* (*Preferred Procedure*).—Weigh 30 g. of the pigment into a 150-ml. beaker and add 50 g. of warm distilled water, freshly boiled to remove carbon dioxide. Mix well by means of a glass rod, cool to 25 C., and determine the pH of the paste or slurry by the standard glass electrode method.

(*2*) *Colorimetric Method* (*Alternate Procedure*) *for Use in Cases Where Glass Electrode is not Available.*—Weigh 10 g. of pigment into a 250-ml. glass-stoppered Erlenmeyer flask (Note 1) and add 150 g. of warm distilled water, freshly boiled to remove carbon dioxide. Stopper the flask and shake vigorously for about 1 min. Cool to 25 C. Let stand 1 hr. (Note 2). Decant and promptly determine the pH of the aqueous portion of the mixture by means of a suitable indicator using the block comparator or similar colorimetric method (Note 3).

NOTE 1.—Use of a glass stopper in the flask is necessary as rubber or cork would affect the pH.

NOTE 2.—Standing is necessary to allow for settling of pigment.

NOTE 3.—Block comparator or other colored solution compensating device should be used as the extract will have a slight color.

Tentative Specifications for

RED AND BROWN IRON OXIDE PIGMENTS[1]

A.S.T.M. Designation: D 84 – 43 T

ISSUED, 1943.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. (*a*) These specifications cover three classes of iron oxide pigments, as follows:

Class I.—Iron oxides commercially pure (including Indian red).

Class II.—Iron oxides containing from 70 to 97 per cent Fe_2O_3.

Class III.—Iron oxides containing from 40 to 70 per cent Fe_2O_3.

(*b*) Classes II and III include two types as follows:

Type A.—Pigments designed to be used when a high sulfate content of the pigment will result in paints of inferior quality, and

Type B.—Pigments designed to be used when a restriction on the sulfate content is unnecessary.

(*c*) Venetian red and earth colors such as siennas and umbers are not covered by these specifications.

Composition and Properties

2. (*a*) *Dry Pigments.*—The pigments shall conform to the requirements for composition as prescribed in Table I.

TABLE I.—REQUIREMENTS FOR IRON OXIDE PIGMENTS.

	Class I	Class II		Class III	
		Type A	Type B	Type A	Type B
Total iron oxide, calculated as Fe_2O_3, min., per cent	97.0	70.0	70.0	40.0	40.0
Moisture and other volatile matter, max., per cent	1.0	2.0	2.0	2.0	2.0
Sulfur calculated as SO_3, max., per cent	2.0	5.0	...	5.0	...
Matter soluble in water, max., per cent	1.0	2.0	2.0	2.0	2.0
Organic coloring matter	none	none	none	none	none
Coarse particles (total residue retained on a No. 325 (44-micron) sieve), max., per cent	1.0	2.0	2.0	2.0	2.0

(*b*) *Paste in Oil.*—The paste shall be made by thoroughly grinding the specified pigment with linseed oil in such proportions as to produce satisfactory consistency. As received it shall not be caked in the container and shall break up readily in linseed oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions without curdling with linseed oil, turpentine, or volatile petroleum spirits, or any mix-

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Accepted by Committee E-10 on Standards, August 30, 1943.

Prior to their present publication as tentative, these specifications were published as tentative from 1921 to 1927, being revised in 1924, 1925, and 1927. They were adopted in 1927, published as standard from 1927 to 1943, being revised in 1940 and 1941, but withdrawn, revised, and republished as tentative in 1943.

ture of these substances. The pastes shall conform to the following requirements:

Coarse particles and skins (total residue retained on a No. 325 (44-micron) sieve), max., per cent of dry pigment	2.5
Moisture and other volatile matter, max., per cent	3

(*c*) Inasmuch as iron oxide pigments are available in a range of colors, the mass color and, if desired by the purchaser, the character of the tint formed by mixture with a white pigment shall be the same as, and the strength not less than, that of a reference sample mutually agreed upon by the seller and the purchaser.

Number of Tests

3. Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10,000 lb., except that for shipments of less than 10,000 lb. two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

Methods of Testing

4. Tests shall be conducted in accordance with the appropriate methods of the American Society for Testing Materials, where applicable. Test procedures not covered by A.S.T.M. methods shall be mutually agreed upon by the seller and the purchaser.

Tentative Specifications for

RAW UMBER[1]

A.S.T.M. Designation: D 763 – 44 T

ISSUED, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These specifications cover the pigment commercially known as raw umber.

Composition and Properties

2. (*a*) The pigment shall be in a soft, dry form and shall be a hydrated oxide of iron (together with such manganese that is naturally associated with it) permeating a siliceous base and shall be free of admixtures of other substances except carbon pigments. The pigment shall conform to the following requirements:[a]

Iron oxide (Fe_2O_3), min., per cent	37.0
Calcium compounds (as CaO), max., per cent	5.0
Moisture and other volatile matter, max., per cent	5.0
Coarse particles (total residue retained on a No. 325 (44-micron) sieve), max., per cent	2.0
Organic colors, max., per cent	none

[a] Genuine manganese umbers not being commercially obtainable at the time of issuing these specifications, a requirement for minimum manganese oxide is omitted, pending return to normal conditions.

(*b*) The mass color and character of tint formed by mixture with a white pigment shall be the same as, and the strength not less than, that of a reference sample mutually agreed upon by the purchaser and the seller.

Number of Tests

3. Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10,000 lb., except that for shipments of less than 10,000 lb. two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

Methods of Testing

4. Tests shall be conducted in accordance with the appropriate methods of the American Society for Testing Materials, where applicable. Tests procedures not covered by A.S.T.M. methods shall be mutually agreed upon by the purchaser and the seller.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Accepted by the Society at annual meeting, June, 1944.

Tentative Specifications for

BURNT UMBER[1]

A.S.T.M. Designation: D 764 – 44 T

ISSUED, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These specifications cover the pigment commercially known as burnt umber.

Composition and Properties

2. (*a*) The pigment shall be produced by the calcination of raw umber and shall be free of admixtures of other substances except carbon pigments. The pigment shall conform to the following requirements:[a]

Iron oxide (Fe_2O_3), min., per cent	42.0
Calcium compounds (as CaO), max., per cent	5.0
Moisture and other volatile matter,[b] max., per cent	5.0
Coarse particles (total residue retained on a No. 325 (44-micron) sieve), max., per cent	2.0
Organic colors, max., per cent	none

[a] Genuine manganese umbers not being commercially obtainable at the time of issuing these specifications, a requirement for minimum manganese oxide is omitted, pending return to normal conditions.

[b] Moisture and other volatile matter shall be loss at 105 C.

Ignition loss shall be the additional loss at temperatures above 105 C.

(*b*) The mass color and character of tint formed by mixture with a white pigment shall be the same as, and the strength not less than, that of a reference sample mutually agreed upon by the purchaser and the seller.

Number of Tests

3. Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10,000 lb., except that for shipments of less than 10,000 lb. two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

Methods of Testing

4. Tests shall be conducted in accordance with the appropriate methods of the American Society for Testing Materials, where applicable. Test procedures not covered by A.S.T.M. methods shall be mutually agreed upon by the purchaser and the seller.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Accepted by the Society at annual meeting, June, 1944.

Tentative Specifications for

RAW SIENNA[1]

A.S.T.M. Designation: D 765 – 44 T

ISSUED, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These specifications cover the pigment commercially known as raw sienna.

Composition and Properties

2. (*a*) The pigment shall be in a soft, dry form and shall be a hydrated oxide of iron permeating a siliceous base and shall conform to the following requirements:

Iron oxide (Fe_2O_3), min., per cent	38.0
Calcium compounds (as CaO), max., per cent	5.0
Moisture and other volatile matter, max., per cent	4.0
Coarse particles (total residue retained on a No. 325 (44-micron) sieve), max., per cent	2.0
Organic colors, max., per cent	none

(*b*) The mass color and character of tint formed by mixture with a white pigment shall be the same as, and the strength not less than, that of a reference sample mutually agreed upon by the purchaser and the seller.

Number of Tests

3. Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10,000 lb., except that for shipments of less than 10,000 lb. two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

Methods of Testing

4. Tests shall be conducted in accordance with the appropriate methods of the American Society for Testing Materials, where applicable. Test procedures not covered by A.S.T.M. methods shall be mutually agreed upon by the purchaser and the seller.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Accepted by the Society at annual meeting, June, 1944.

Tentative Specifications for

BURNT SIENNA[1]

A.S.T.M. Designation: D 766 – 44 T

ISSUED, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These specifications cover the pigment commercially known as burnt sienna.

Composition and Properties

2. (*a*) The pigment shall be produced by the calcination of raw sienna and shall conform to the following requirements:

Iron oxide (Fe_2O_3), min., per cent	40.0
Calcium compounds (as CaO), max., per cent	5.0
Moisture and other volatile matter, max., per cent	4.0
Coarse particles (total residue retained on a No. 325 (44-micron) sieve), max., per cent	2.0
Organic colors, max., per cent	none

(*b*) The mass color and character of tint formed by mixture with a white pigment shall be the same as, and the strength not less than, that of a reference sample mutually agreed upon by the purchaser and the seller.

Number of Tests

3. Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10,000 lb., except that for shipments of less than 10,000 lb. two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

Methods of Testing

4. Tests shall be conducted in accordance with the appropriate methods of the American Society for Testing Materials, where applicable. Test procedures not covered by A.S.T.M. methods shall be mutually agreed upon by the purchaser and the seller.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Accepted by the Society at annual meeting, June, 1944.

Tentative Specifications for
VENETIAN RED[1]

A.S.T.M. Designation: D 767 – 44 T

ISSUED, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These specifications cover the pigment commercially known as venetian red.

Composition and Properties

2. (*a*) The pigment shall be in a soft, dry form and shall consist of iron oxide (Fe_2O_3) on a base consisting essentially of calcium sulfate and shall consist of three main types I, II, and III, which shall conform to the following requirements:

	Type I	Type II	Type III
Ferric oxide, min., per cent	40.0	30.0	20.0
Moisture and other volatile matter, max., per cent	4.0	4.0	4.0
Coarse particles (total residue retained on a No. 325 (44-micron) sieve), max., per cent	2.0	2.0	2.0
Loss on ignition,[a] max., per cent	1.0	1.0	1.0
Calcium carbonate ($CaCO_3$), max., per cent	0.1	0.1	0.1
Hydrogen ion concentration (pH value)	8.0 max.	6.0 min.	....

[a] Loss on ignition shall be calculated on the dry material.

(*b*) The mass color and character of tint formed by mixture with a white pigment shall be the same as, and the strength not less than, that of a reference sample mutually agreed upon by the purchaser and the seller.

Number of Tests

3. Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10,000 lb., except that for shipments of less than 10,000 lb. two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

Methods of Testing

4. (*a*) *General.*—Tests shall be conducted in accordance with the appropriate methods of the American Society for Testing Materials, where applicable. Test procedures not covered by A.S.T.M. methods shall be mutually agreed upon by the purchaser and the seller.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Accepted by the Society at annual meeting, June, 1944.

(*b*) *Hydrogen Ion Concentration Test.*—Hydrogen ion concentration shall be determined in accordance with either of the following methods:

(*1*) *Electrometric Method (Preferred Procedure).*—Weigh 30 g. of the pigment into a 150-ml. beaker and add 50 g. of warm distilled water, freshly boiled to remove carbon dioxide. Mix well by means of a glass rod, cool to 25 C., and determine the pH of the paste or slurry by the standard glass electrode method.

(*2*) *Colorimetric Method (Alternate Procedure) for Use in Cases Where Glass Electrode is not Available.*—Weigh 10 g. of pigment into a 250-ml. glass-stoppered Erlenmeyer flask (Note 1) and add 150 g. of warm distilled water, freshly boiled to remove carbon dioxide. Stopper the flask and shake vigorously for about 1 min. Cool to 25 C. Let stand 1 hr. (Note 2). Decant and promptly determine the pH of the aqueous portion of the mixture by means of a suitable indicator using the block comparator or similar colorimetric method (Note 3).

NOTE 1.—Use of a glass stopper in the flask is necessary as rubber or cork would affect the pH.

NOTE 2.—Standing is necessary to allow for settling of pigment.

NOTE 3.—Block comparator or other colored solution compensating device should be used as the extract will have a slight color.

Tentative Specifications for

YELLOW IRON OXIDE, HYDRATED[1]

A.S.T.M. Designation: D 768 - 44 T

ISSUED, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These specifications cover the pigment commercially known as yellow iron oxide, hydrated.

Composition and Properties

2. (*a*) The pigment shall be a manufactured yellow iron oxide obtained by chemical reaction. It shall be a soft, finely disintegrated pigment, free of admixtures of other substances and shall conform to the following requirements:

Total hydrated oxide of iron, min., per cent	97.0
Total oxide of iron,[a] min., per cent	86.0
Loss on ignition,[a] max., per cent	13.0
Moisture and other volatile matter, max., per cent	1.0
Water soluble material, max., per cent	1.0
Coarse particles (total residue retained on a No. 325 (44-micron) sieve), max., per cent	0.5
Hydrogen ion concentration (pH Value)	5.5 to 7.5

[a] Total hydrated oxide of iron shall be the sum of iron oxide and loss on ignition. Loss on ignition shall be calculated on the dry material.

(*b*) The mass color and character of tint formed by mixture with a white pigment shall be the same as, and the strength not less than, that of a reference sample mutually agreed upon by the purchaser and the seller.

Number of Tests

3. Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10,000 lb., except that for shipments of less than 10,000 lb. two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

Methods of Testing

4. (*a*) *General.*—Tests shall be conducted in accordance with the appropriate methods of the American Society for Testing Materials, where applicable. Test procedures not covered by A.S.T.M.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Accepted by the Society at annual meeting, June, 1944.

methods shall be mutually agreed upon by the purchaser and the seller.

(*b*) *Hydrogen Ion Concentration Test.*—Hydrogen ion concentration shall be determined in accordance with either of the following methods:

(*1*) *Electrometric Method* (*Preferred Procedure*).—Weigh 30 g. of the pigment into a 150-ml. beaker and add 50 g. of warm distilled water, freshly boiled to remove carbon dioxide. Mix well by means of a glass rod, cool to 25 C., and determine the pH of the paste or slurry by the standard glass electrode method.

(*2*) *Colorimetric Method* (*Alternate Procedure*) *for Use in Cases Where Glass Electrode is not Available.*—Weigh 10 g. of pigment into a 250-ml. glass-stoppered Erlenmeyer flask (Note 1) and add 150 g. of warm distilled water, freshly boiled to remove carbon dioxide. Stopper the flask and shake vigorously for about 1 min. Cool to 25 C. Let stand 1 hr. (Note 2). Decant and promptly determine the pH of the aqueous portion of the mixture by means of a suitable indicator using the block comparator or similar colorimetric method (Note 3).

NOTE 1.—Use of a glass stopper in the flask is necessary as rubber or cork would affect the pH.

NOTE 2.—Standing is necessary to allow for settling of pigment.

NOTE 3.—Block comparator or other colored solution compensating device should be used as the extract will have a slight color.

Tentative Methods of

ANALYSIS OF BARIUM SULFATE PIGMENTS[1]

A.S.T.M. Designation: D 715 - 43 T

ISSUED, 1943.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These methods cover the procedures for the chemical analysis of barium sulfate pigments.

BARIUM SULFATE

Procedure

2. (*a*) Transfer 0.5000 g. of the sample to a platinum crucible, add 3 g. of Na_2CO_3, mix thoroughly, and fuse until the melt is clear. Allow the melt to cool, and then leach in a platinum dish with hot water until it is entirely disintegrated. Filter on a close-texture paper,[3] and wash thoroughly with hot Na_2CO_3 (30 g. per l.).

(*b*) Transfer the filter paper containing the insoluble carbonates to a 250-ml. beaker, and acidify with HCl (sp. gr. 1.18). Wash the fusion crucible with HCl (sp. gr. 1.18) so that no barium is lost. Boil the solution, filter into a 600-ml. beaker, and wash well with water. Add methyl red indicator, and add NH_4OH (sp. gr. 0.90) until the solution is slightly alkaline. Add 6 ml. of HCl (1:1), and dilute to 300 ml.

(*c*) Heat the solution to boiling, and add 5 g. of $(NH_4)_2SO_4$ dissolved in 40 ml. of water. If low-grade material is being analyzed, the $(NH_4)_2SO_4$ solution should be added drop by drop from a burette to minimize inclusion. If the barytes is a rather pure product (95 to 99 per cent $BaSO_4$) this is not necessary, since the only nonvolatile constituents of the solution will be barium salts. Allow the precipitate of $BaSO_4$ to digest for 4 hr. or overnight, and filter through a close-texture paper.[3] Wash the precipitate with as little cold water as possible (consistent with the purity of the precipitate), ignite in an oxidizing atmosphere, and weigh as $BaSO_4$.

(*d*) *Calculation.*—Calculate the percentage of $BaSO_4$ as follows:

$$BaSO_4, \text{ per cent} = \frac{\text{wt. of } BaSO_4}{\text{wt. of sample}} \times 100$$

FERRIC OXIDE

Apparatus

3. *Colorimetric Apparatus.*—Nessler

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Accepted by the Society at annual meeting, June, 1943.

[3] No 602 S & S blue ribbon or Whatman No. 2 filter paper is suitable for this purpose.

type or other similar 100-ml. colorimetric tubes will be required.

Reagents

4. (*a*) *Potassium Permanganate Solution (0.1 g. per l.).*

(*b*) *Ammonium Thiocyanate Solution (76.1 g. per l., 1 N).*

(*c*) *Standard Iron Solution (100 ml. = 0.00002 g. Fe).*—Dilute and divide a ferric solution of known iron content so as to obtain 0.0004 g. of iron. Dilute the solution to 2 l. with water containing 200 ml. of iron-free, c.p. H_2SO_4.

(*d*) *Standard Color Solution.*—Mix thoroughly 10 parts by volume of ammonium thiocyanate (76.1 g. per l.) and 90 parts by volume of standard iron solution (100 ml. = 0.00002 g. Fe). One hundred milliliters of the solution will thus contain 0.000018 g. of Fe.

Procedure

5. (*a*) Dissolve the soluble portion of a 1-g. sample in H_2SO_4 (1:1) filter, and wash, keeping the volume of the solution under 100 ml. Oxidize any iron present in the filtrate by adding $KMnO_4$ (0.1 g. per l.) until a faint pink color is obtained. Dilute the solution to 100 ml. and pour into a burette graduated in 0.1-ml. divisions.

(*b*) Pour 100 ml. of the standard color solution into a 100-ml. colorimetric tube. Into a second colorimetric tube pour 10 ml. of H_2SO_4 (sp. gr. 1.84) and 10 ml. of NH_4CNS (76.1 g. per l.), dilute to 60 or 70 ml. and then add the sample solution from the burette until the depth of color thus produced on dilution to 100 ml. exactly matches that of the standard. Record the number of milliliters required.

(*c*) *Calculation.*—Calculate the percentage of Fe_2O_3 as follows:

$$Fe_2O_3, \text{ per cent} = \frac{A \times 1.4298}{B} \times 100$$

where:

A = grams of iron in standard, and

B = grams of sample used.

Hydrogen Ion Concentration

Procedure

6. Transfer 25 g. of the sample to a 250 ml. beaker, add 100 ml. of water, and stir for 30 min. Allow the solid material to settle, and determine the pH by any suitable method, preferably with a glass electrode potentiometer (Note).

Note.—The determination of pH is considered preferable to the determination of free acid by titration with standard alkali.

Matter Soluble in Water

Procedure

7. (*a*) Transfer 2.5 g. of the sample to a graduated 250-ml. flask, add 100 ml. of water, boil for 5 min., cool, dilute to 250 ml., mix, and allow to settle. Filter the supernatant liquid through a dry paper and discard the first 20 ml. Evaporate 100 ml. of the clear filtrate to dryness in a weighed dish, heat for 1 hr. at 105 to 110 C., cool, and weigh.

(*b*) *Calculation.*—Calculate the percentage of matter soluble in water as follows:

$$\text{Matter soluble in water, per cent} = \frac{A \times 2.5}{B} \times 100$$

where:

A = grams of residue after drying, and

B = grams of sample used.

Moisture and Other Volatile Matter

Procedure

8. (*a*) Transfer 1 g. of the sample to a wide-mouth, short weighing bottle provided with a glass stopper. Heat, with the stopper removed, for 2 hr. at 105 ± 2 C., insert the stopper, cool, and weigh.

(*b*) *Calculation.*—Calculate the percentage moisture and other volatile matter as follows:

$$\text{Moisture and other volatile matter, per cent} = \frac{\text{wt. loss on heating}}{\text{wt. of sample}} \times 100$$

COARSE PARTICLES

(*Total Residue Retained on No. 325 (44-micron) Sieve*)

Apparatus

9. *Sieve.*—A No. 325 (44-micron) sieve conforming to the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials[4] shall be used.

Procedure

10. (*a*) Dry a No. 325 (44-micron) sieve in an oven at 105 to 110 C., cool, and weigh accurately. Transfer 10 g. of the sample to a mortar, moisten with alcohol, add 100 ml. of water, thoroughly mix by gentle pressure with the finger to break up all lumps, and wash through the sieve with a gentle stream of water, brushing gently with a camel's hair brush until nothing more passes through the sieve. Dry the sieve for 1 hr. at 105 to 110 C., cool, and weigh.

(*b*) *Calculation.*—Calculate the percentage of coarse particles as follows:

$$\text{Coarse particles, per cent} = \frac{\text{wt. increase of sieve}}{\text{wt. of sample}} \times 100$$

FREE SILICA

Reagent

11. (*a*) *Heavy Liquid (sp. gr. 2.9).*—Dissolve 4 parts by weight of KI in 6 parts of warm water, and add, while stirring constantly, 5 parts of HgI_2. Evaporate over a hot plate or gas flame, protected by screen cloth, until a light crystalline scum forms. Cool, and filter through heavy paper. The solution will be of a clear, deep, amber color of about 3.2 specific gravity. All filter washings showing a yellow color should be saved and evaporated. Determine the 3.2 specific gravity accurately by means of a specific gravity bottle, and correct the solution to a specific gravity of 2.9 at room temperature by adding water. Prepare approximately 200 ml. of the solution.

Procedure

12. (*a*) Carefully weigh 10 g. of the finely ground dry barium sulfate pigment and transfer to a carefully dried 125-ml. clear glass separatory funnel of the long narrow type. Add 100 ml. of the heavy liquid (sp. gr. 2.9), stopper, and shake well to liberate any air bubbles which might adhere to the particles, and also to break up all agglomerates. Set the separatory funnel aside and allow to stand in a perpendicular position for 2 hr., or for a sufficient time for the particles to rise or sink.

(*b*) Draw off the heavy particles through the stopcock into a beaker along with 75 ml. of the liquid. Add 50 ml. of fresh heavy liquid (sp. gr. 2.9) to the float particles and liquid remaining in the separatory funnel and repeat the procedure as described in Paragraph (*a*).

(*c*) Draw off the heavy particles along with 75 ml. of the liquid and add to those drawn off in accordance with Paragraph (*a*), leaving the float particles in the funnel. Wash the particles remaining in the funnel with fresh heavy liquid (sp. gr. 2.9), filter through a No. 00 paper, and wash free of solution with warm water.

(*d*) Transfer the filter paper and silica to an ignition cup, dry, and ignite carefully in an electric furnace or over a

[4] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

flame to keep mechanical loss at a minimum. Weigh the floated material, and transfer to a tared platinum dish or crucible. Add 20 ml. of HF and 3 drops of H_2SO_4 (1:1), evaporate to dryness, and ignite carefully to expel all fumes. Cool and weigh. The loss in weight represents the free silica in the floated material.

(*e*) *Calculation.*—Calculate the percentage of free SiO_2 as follows:

$$\text{Free } SiO_2, \text{ per cent} = \frac{\text{wt. } SiO_2}{\text{wt. of sample}} \times 100$$

Tentative Methods of
ANALYSIS OF MICA PIGMENT[1]

A.S.T.M. Designation: D 716 – 43 T

ISSUED, 1943.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These methods cover the procedures for the chemical analysis of mica pigments.

APPARENT DENSITY

Apparatus

2. *Volumeter.*—A Scott volumeter or similar apparatus shall be used modified as follows: The screen used shall conform to the requirements of a No. 40 (420-micron) sieve as prescribed in the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials.[3] The funnel below the screen shall be replaced by a conical funnel having a bottom opening 1 in. in diameter. It may be found necessary to replace the top glass baffle with one that is longer to insure all of the sifted mica being caught.

Procedure

3. (*a*) Transfer convenient quantities of the mica pigment to the funnel of the modified volumeter, and brush the pigment through the screen with a camel's hair brush until the receiver is slightly more than full. Scrape off the excess, and weigh the pigment. Care must be taken not to jar the apparatus during the procedure.

(*b*) *Calculation.*—Calculate the apparent density of the mica pigment and convert to pounds per cubic foot.

Report

4. The average of three determinations shall be reported as the apparent density of the mica pigment.

Reproducibility of Results

5. The average of the three determinations shall check within 5 per cent.

MOISTURE AND OTHER VOLATILE MATTER

Procedure

6. (*a*) Transfer 1 g. of the sample to a wide-mouth, short weighing bottle provided with a glass stopper. Heat, with the stopper removed, for 2 hr. at 105 ± 2 C., insert the stopper, cool, and weigh.

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Accepted by the Society at annual meeting, June, 1943.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(*b*) *Calculation.*—Calculate the percentage moisture and other volatile matter as follows:

$$\text{Moisture and other volatile matter, per cent} = \frac{\text{wt. loss on heating}}{\text{wt. of sample}} \times 100$$

GRIT

Apparatus

7. (*a*) *Beaker.*—A 600-ml. low-form beaker shall be used.

(*b*) *Metal Tubing.*—An L-shape metal tube (see Fig. 1) $\frac{1}{4}$ in. in outside diameter

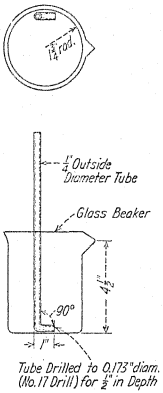


FIG. 1.—Grit Test Apparatus.

with a foot 1 in. in length. The foot of the tube shall be drilled to 0.173 in. (No. 17 drill) in inside diameter for a depth of $\frac{1}{2}$ in. The tube shall be placed in the beaker with the long arm in the vertical position, and the foot of the L parallel to the bottom of the beaker and perpendicular to the radius of the bottom at a point such that there is about $\frac{1}{16}$-in. clearance between the tubing and both the bottom and side of the beaker. The center of the foot shall be placed 90 deg. from the lip of the beaker.

Procedure

8. (*a*) Transfer 10 g. of the sample to the 600-ml. beaker, add 100 ml. of water, and swirl the beaker until the mica is dispersed. Insert the metal tubing into the beaker as described in Section 7 (*b*) and connect the upper end to a water faucet. Regulate the flow of water by a constant head to a rate of 800 ml. per min. over the lip of the beaker. When the elutriation has progressed so that the body of the water in the beaker is clear, stop the flow, and lower the water level to prevent spilling.

(*b*) Swirl the beaker again to bring the remaining mica pigment into suspension and repeat the elutriation procedure as described in Paragraph (*a*) three times. After final clearing, decant part of the water, and filter the residue. Transfer the paper and residue to a weighed crucible, and ash slowly, cool, and weigh.

(*c*) *Calculation.*—Calculate the percentage of grit as follows:

$$\text{Grit, per cent} = \frac{(\text{wt. of ash}) - (\text{wt. of filter paper ash})}{\text{wt. of sample}} \times 100$$

COARSE PARTICLES

Apparatus

9. *Sieve.*—A No. 140 (105-micron) and a No. 325 (44-micron) sieve conforming to the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials[3] shall be used.

Procedure

10. (*a*) *Total Residue Retained on a No. 140 (105-micron) Sieve.*—Dry a No. 140 (105-micron) sieve in an oven at 105 to 110 C., cool, and weigh accurately.

Transfer 10 g. of the sample to a mortar, moisten with alcohol, add 100 ml. of water, thoroughly mix by gentle pressure with the finger to break up all lumps, and wash through the sieve with a gentle stream of water, brushing gently with a camel's hair brush until nothing more passes through the sieve. Dry the sieve for 1 hr. at 105 to 110 C., cool, and weigh.

(*b*) *Calculation.*—Calculate the percentage of coarse particles as follows:

$$\text{Coarse particles, per cent} = \frac{\text{wt. increase of sieve}}{\text{wt. of sample}} \times 100$$

(*c*) *Total Residue Retained on a No. 325 (44-Micron) Sieve.*—Determine the coarse particles as described in Paragraphs (*a*) and (*b*) using a 1-g. sample and substituting a No. 325 (44-micron) sieve.

COLOR

Standard Pigments

11. (*a*) *Standard Mica Pigment.*
(*b*) *Standard Zinc Oxide..*

Procedure

12. Compare pastes made by rubbing a standard mica pigment and standard zinc oxide, mutually agreed upon by the purchaser and the seller, in linseed oil with a similar paste using the sample to be tested. Rub 9 g. of zinc oxide, and 3 g. of mica pigment on a glass plate with a glass muller or rubber spatula to avoid staining, with 4 ml. of bleached linseed oil. Spread the pastes adjacently on a clear glass plate and determine the color by viewing the spreadouts on the glass.

Tentative Methods of
ANALYSIS OF MAGNESIUM SILICATE PIGMENT[1]

A.S.T.M. Designation: D 717 – 43 T

ISSUED, 1943.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These methods cover the procedures for the chemical analysis of magnesium silicate pigment.

SILICON DIOXIDE

Procedure

2. (*a*) Transfer 1 g. of the sample to a platinum crucible and fuse with 5 g. of Na_2CO_3, starting the fusion over a small flame, then increase the heat gradually until the entire contents of the crucible are in a molten state, and continue heating for 20 min. Keep a close-fitting platinum cover on the crucible during the fusion. When the fusion is complete, allow the crucible and contents to cool, and transfer to a 600-ml. porcelain casserole containing 200 ml. of water (Note). Boil until the melt is disintegrated.

NOTE.—If, during the cooling period, the crucible is partially immersed several times in cold water to chill the outer portions of the melt, the subsequent removal of the melt is facilitated. Do not allow the water to enter the crucible while the contents are hot, because of possible spattering.

(*b*) Remove the crucible and lid being careful to scrub and rinse out any adhering particles of the melt. Carefully acidify the contents of the casserole with HCl (sp. gr. 1.18); introduce the HCl (sp. gr. 1.18) in small portions, keeping a watch glass over the crucible in order to avoid loss by spattering. Add 30 ml. of HCl (sp. gr. 1.18) in excess and evaporate to dryness on a steam bath, care being taken to break up any crusts which form. When the material appears completely dry, and no odor of HCl can be detected, remove the casserole from the steam bath, and allow to cool.

(*c*) Wash down the sides of the casserole with 20 ml. of HCl (sp. gr. 1.18) and then with water. Repeat the evaporation as described in Paragraph (*b*), then bake for 1 hr. in an oven at 105 C. Cool the residue, drench with 25 ml. of HCl (sp. gr. 1.18), add 175 ml. of water, and warm, while stirring, until all soluble salts are dissolved. Filter off the silica on a close-texture paper, wash five times with HCl (1:20), wash five times with hot water, and

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Accepted by the Society at annual meeting, June, 1943.

reserve the filtrate for the determination of magnesium oxide (Section 3).

(*d*) Transfer the paper and washed silica to a clean platinum crucible, ignite, first gently until the filter paper is consumed, and then at 1200 C. for 20 min., cool, and weigh. Moisten the residue with water, add 5 drops of H_2SO_4 (sp. gr. 1.84), and 15 ml. of HF. Evaporate to dryness on a steam bath, heat gently until H_2SO_4 has been expelled, and ignite at 1200 C. for 5 min. Cool and weigh. The loss in weight represents the SiO_2.

(*e*) *Calculation.*—Calculate the percentage of silica as follows:

$$SiO_2, \text{ per cent} = \frac{\text{wt. of } SiO_2}{\text{wt. of sample}} \times 100$$

MAGNESIUM OXIDE

Procedure

3. (*a*) If an appreciable residue remains after the treatment with HF in accordance with Section 2 (*d*), fuse the residue with a small amount of $K_4S_2O_7$ until it is dissolved. Leach the pyrosulfate melt out of the crucible with water and combine the solution with the filtrate reserved in accordance with Section 2 (*c*).

(*b*) Remove the iron and aluminum from the combined solutions by precipitation with NH_4OH and filtration in the usual manner, and follow with a double precipitation of calcium in the alkaline filtrate by ammonium oxalate in the usual manner.[8]

(*c*) Acidify slightly the combined filtrates from the calcium precipitation (which should be adjusted to a volume of 400 ml.) with HCl, and add a ten-fold excess of $(NH_4)_2HPO_4$. If the solution becomes alkaline, add HCl (1:1) to keep the solution acidic. When the $(NH_4)_2HPO_4$ has completely dissolved, add NH_4OH (1:3) slowly, while stirring, until a precipitate begins to appear; this should occur at the time the solution becomes alkaline and can best be induced by rapid stirring and rubbing the sides of the beaker with the glass stirring rod. Continue stirring, and add NH_4OH (1:3) until no further precipitation occurs. Add 50 ml. of NH_4OH (sp. gr. 0.90), and allow the solution to stand overnight.

(*d*) Filter the solution on a close-texture paper, and wash with NH_4OH (5:95). Carefully dissolve the magnesium ammonium phosphate precipitate by alternately pouring through the filter HCl (1:3) and water. Catch the filtrate in a clean 400-ml. beaker, adjust the volume to 250 ml., and add 0.2 g. of $NH_4H_2PO_4$. Add NH_4OH (1:3) slowly, while stirring rapidly, until a precipitate begins to appear, and continue stirring until precipitation is apparently complete. Add 25 ml. of NH_4OH (sp. gr. 0.90) and allow to stand for 4 hr.

(*e*) Filter the solution on a close-texture paper, and wash ten times with NH_4OH (5:95). Transfer the precipitate wrapped in the moist paper to a weighed platinum crucible, char the paper slowly without allowing it to ignite, and burn the carbon off over a gradually increased flame which should never heat the crucible to more than the faintest red. Finally heat at 1000 to 1100 C. for 30 min., cool in a desiccator, and weigh as $Mg_2P_2O_7$. Repeat the heating to constant weight.

(*f*) *Calculation.*—Calculate the percentage of MgO as follows:

$$MgO, \text{ per cent} = \frac{\text{wt. of } Mg_2P_2O_7 \times 0.3621}{\text{wt. of sample}} \times 100$$

[8] Reference may be made to the procedures described in Sections 9 and 13 of the Standard Methods of Chemical Analysis of Portland Cement (A.S.T.M. Designation: C 114), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

LOSS ON IGNITION

Procedure

4. (*a*) Transfer an accurately weighed portion of the sample (about 1 g.) to a previously ignited, weighed porcelain crucible, and ignite at 900 to 1000 C. for 20 min. Cool in a desiccator, and weigh. Heat again for 5 min. to check the loss in weight.

(*b*) *Calculation.*—Calculate the percentage loss on ignition as follows:

$$\text{Loss on ignition, per cent} = \frac{\text{wt. decrease of sample after ignition}}{\text{wt. of sample}} \times 100$$

MOISTURE AND OTHER VOLATILE MATTER

Procedure

5. (*a*) Transfer 1 g. of the sample to a wide-mouth, short weighing bottle provided with a glass stopper. Heat, with the stopper removed, for 2 hr. at 105 ± 2 C., insert the stopper, cool, and weigh.

(*b*) *Calculation.*—Calculate the percentage moisture and other volatile matter as follows:

$$\text{Moisture and other volatile matter, per cent} = \frac{\text{wt. loss on heating}}{\text{wt. of sample}} \times 100$$

COARSE PARTICLES

(*Total Residue Retained on No. 325 (44-micron) Sieve*)

Apparatus

6. *Sieve.*—A No. 325 (44-micron) sieve conforming to the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials,[4] shall be used.

[4] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Procedure

7. (*a*) Dry a No. 325 (44-micron) sieve in an oven at 105 to 110 C., cool, and weigh accurately. Transfer 10 g. of the sample to a mortar, moisten with alcohol, add 100 ml. of water, thoroughly mix by gentle pressure with the finger to break up all lumps, and wash through the sieve with a gentle stream of water, brushing gently with a camel's hair brush until nothing more passes through the sieve. Dry the sieve for 1 hr. at 105 to 110 C., cool, and weigh.

(*b*) *Calculation.*—Calculate the percentage of coarse particles as follows:

$$\text{Coarse particles, per cent} = \frac{\text{wt. increase of sieve}}{\text{wt. of sample}} \times 100$$

COLOR

Standard Pigment

8. (*a*) *Standard Extender Pigment.*
(*b*) *Standard Zinc Oxide.*

Procedure

9. Carefully weigh out the proportions of the standard extender pigment and standard zinc oxide mutually agreed upon, and rub up to a fairly stiff paste with a glass muller on a glass plate or stone slab with raw linseed oil conforming to the Standard Specifications for Raw Linseed Oil (A.S.T.M. Designation: D 234) of the American Society for Testing Materials.[4] Note the volume of the oil required. Prepare a similar paste with the sample, using the same weight of pigment, volume of oil, and number of strokes of the glass muller as used in the preparation of the paste of the standard pigments. Spread the pastes adjacently on a microscope slide, draw a scraper lightly across them so as to present them on an even plane, and judge the color immediately.

Tentative Methods of
ANALYSIS OF ALUMINUM SILICATE PIGMENT[1]

A.S.T.M. Designation: D 718 - 43 T

ISSUED, 1943.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These methods cover the procedures for the chemical analysis of aluminum silicate pigment.

SILICON DIOXIDE

Procedure

2. (*a*) Transfer 1 g. of the sample to a platinum crucible and fuse with 5 g. of Na_2CO_3, starting the fusion over a small flame, then increase the heat gradually until the entire contents of the crucible are in a molten state, and continue heating for 20 min. Keep a close-fitting cover on the crucible during the fusion. When the fusion is complete, allow the crucible and contents to cool, and transfer to a 600-ml. porcelain casserole containing 200 ml. of water (Note). Boil until the melt is disintegrated.

NOTE.—If, during the cooling period, the crucible is partially immersed several times in cold water to chill the outer portions of the melt, the subsequent removal of the melt is facilitated. Do not allow the water to enter the crucible while the contents are hot, because of possible spattering.

(*b*) Remove the crucible and lid being careful to scrub and rinse out any adhering particles of the melt. Carefully acidify the contents of the casserole with HCl (sp. gr. 1.18); introduce the HCl (sp. gr. 1.18) in small portions, keeping a watch glass over the crucible in order to avoid loss by spattering. Add 30 ml. of HCl (sp. gr. 1.18) in excess and evaporate to dryness on a steam bath, care being taken to break up any crusts which form. When the material appears completely dry, and no odor of HCl can be detected, remove the casserole from the steam bath, and allow to cool.

(*c*) Wash down the sides of the casserole with 20 ml. of HCl (sp. gr. 1.18) and then with water. Repeat the evaporation as described in Paragraph (*b*), then bake for 1 hr. in an oven at 105 C. Cool the residue, drench with 25 ml. of HCl (sp. gr. 1.18), add 175 ml. of water, and warm, while stirring, until all soluble salts are dissolved. Filter off the silica on a close-texture paper, wash five times with HCl (1:20), wash five times with

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Accepted by the Society at annual meeting, June, 1943.

hot water, and reserve the filtrate for the determination of aluminum oxide (Section 4).

(*d*) Transfer the paper and washed silica to a clean platinum crucible, ignite, first gently until the filter paper is consumed, and then at 1200 C. for 20 min., cool, and weigh. Moisten the residue with water, add 5 drops of H_2SO_4 (sp. gr. 1.84), and 15 ml. of HF. Evaporate to dryness on a steam bath, heat gently until H_2SO_4 has been expelled, and ignite at 1200 C. for 5 min. Cool and weigh. The loss in weight represents the SiO_2.

(*e*) *Calculation.*—Calculate the percentage of silica as follows:

$$SiO_2, \text{ per cent} = \frac{\text{wt. of } SiO_2}{\text{wt. of sample}} \times 100$$

ALUMINUM OXIDE

Reagents

3. (*a*) *Stannous Chloride Solution.*—Dissolve 5 g. of $SnCl_2 \cdot 2H_2O$ in 10 ml. of HCl (sp. gr. 1.18), and dilute to 100 ml. with water. Add scraps of iron-free granulated tin, and boil until the solution is clear. Keep the solution in a closed dropping bottle containing metallic tin.

(*b*) *Sulfuric - Phosphoric Acid Mixture.*—Add slowly, while stirring, 150 ml. of H_3PO_4 (sp. gr. 1.71, 85 to 88 per cent) and 150 ml. of H_2SO_4 (sp. gr. 1.84) to 500 ml. of water, dilute to 1 liter with water, and mix.

(*c*) *Diphenylamine Indicator Solution.*—Dissolve 1 g. of diphenylamine in 100 ml. of H_2SO_4 (sp. gr. 1.84).

(*d*) *Mercuric Chloride Solution (Saturated).*

(*e*) *Standard Potassium Dichromate Solution (0.05 N).*—Dissolve 2.457 g. of $K_2Cr_2O_7$ in water and dilute to 1 liter. Standardize against National Bureau of Standards' standard sample No. 27*b* of Sibley iron ore, using such an amount as to give approximately the same titration as the sample to be analyzed.

Procedure

4. (*a*) If an appreciable residue remains after the treatment with HF in accordance with Section 2 (*d*), fuse the residue with a small amount of $K_4S_2O_7$ until it is dissolved. Leach the pyrosulfate melt out of the crucible with water and combine the solution with the filtrate reserved in accordance with Section 2 (*c*).

(*b*) Bring the volume of the combined solution to 250 ml., and, if necessary, add HCl in order to insure a total of 10 to 15 ml. of HCl, add a few drops of methyl red indicator solution, and heat to boiling. Add NH_4OH (sp. gr. 0.90) drop by drop until 1 drop changes the color of the solution to a distinct yellow. Reheat the solution containing the precipitated hydroxides to boiling, boil for 1 or 2 min., and filter. Wash the precipitate once by decantation and then slightly on the filter with hot NH_4Cl (20 g. per 1.).

(*c*) Transfer the precipitate and paper to the original beaker in which the precipitation was made. Dissolve the precipitate in hot HCl (1:3), dilute to 100 ml., and precipitate again as described in Paragraph (*b*). After filtering, wash the precipitate ten times with small portions of hot NH_4Cl (20 g. per 1.). Transfer the precipitate to a weighed platinum crucible, heat slowly until the paper is charred, and finally ignite to constant weight at 1050 to 1100 C. in an electric furnace or over a burner, care being taken to avoid reduction. Weigh the precipitate as $Al_2O_3 + Fe_2O_3$.

(*d*) Determine the percentage of Fe_2O_3 alone as follows and subtract from the percentage of $Al_2O_3 + Fe_2O_3$ to obtain the percentage of Al_2O_3: Transfer a separate sample of 1 g. to a 250-ml. beaker, add 20 ml. of cold water, stir, and add 10 ml. of HCl

(sp. gr. 1.18). Warm, and grind the sample with the flattened end of a glass rod until decomposition is complete. Dilute the solution to 50 ml., heat to boiling, and add 5 ml. of HCl (sp. gr. 1.18). Add $SnCl_2$ solution drop by drop, while stirring, until the solution is decolorized, and then add 1 drop more. Cool the solution to room temperature, wash down the inside of the beaker with water, and add at one stroke 10 ml. of cool $HgCl_2$ solution (saturated). Stir the solution vigorously for 1 min., add 15 ml. of H_2SO_4-H_3PO_4 mixture, and then add 3 drops of diphenylamine indicator solution. Dilute the solution to 150 ml. with cold water. Titrate with 0.05 *N* $K_2Cr_2O_7$ to an intense deep-blue end point that remains unchanged on further addition of 0.05 *N* $K_2Cr_2O_7$.

(*c*) *Calculation.*—Calculate the percentage of Al_2O_3 as follows:

$$Al_2O_3 + Fe_2O_3, \text{ per cent} = \frac{A}{B} \times 100$$

$$Fe_2O_3, \text{ per cent} = \frac{DC \times 0.0798}{B} \times 100$$

Al_2O_3, per cent =

$(Al_2O_3 + Fe_2O_3$, per cent$) - Fe_2O_3$, per cent

where:

A = weight of $Al_2O_3 + Fe_2O_3$,
B = grams of sample used,
C = milliliters of $K_2Cr_2O_7$ required to titrate the sample, and
D = normality of the $K_2Cr_2O_7$.

Loss on Ignition

Procedure

5. (*a*) Transfer an accurately weighed portion of the sample (about 1 g.) to a previously ignited, weighed porcelain crucible, and ignite at 900 to 1000 C. for 20 min. Cool in a desiccator, and weigh. Heat again for 5 min. to check the loss in weight.

(*b*) *Calculation.*—Calculate the percentage loss on ignition as follows:

$$\text{Loss on ignition, per cent} = \frac{\text{wt. decrease of sample after ignition}}{\text{wt. of sample}} \times 100$$

Moisture and Other Volatile Matter

Procedure

6. (*a*) Transfer 1 g. of the sample to a wide-mouth short weighing bottle provided with a glass stopper. Heat, with the stopper removed, for 2 hr. at 105 ± 2 C., insert the stopper, cool, and weigh.

(*b*) *Calculation.*—Calculate the percentage of moisture and other volatile matter as follows:

$$\text{Moisture and other volatile matter, per cent} = \frac{\text{wt. loss on heating}}{\text{wt. of sample}} \times 100$$

Coarse Particles

(*Total Residue Retained on a No. 325 (44-micron) Sieve*)

Apparatus

7. *Sieve.*—A No. 325 (44-micron) sieve conforming to the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials,[3] shall be used.

Procedure

8. (*a*) Dry a No. 325 (44-micron) sieve in an oven at 105 to 110 C., cool, and weigh accurately. Transfer 10 g. of the sample to a mortar, moisten with alcohol, add 100 ml. of water, thoroughly mix by gentle pressure with the finger to break up all lumps, and wash through the sieve with a gentle stream of water,

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

brushing gently with a camel's hair brush until nothing more passes through the sieve. Dry the sieve for 1 hr. at 105 to 110 C., cool, and weigh.

(*b*) *Calculation.*—Calculate the percentage of coarse particles as follows:

$$\text{Coarse particles, per cent} = \frac{\text{wt. increase of sieve}}{\text{wt. of sample}} \times 100$$

COLOR

Standard Pigment

9. (*a*) *Standard Extender Pigment.*
(*b*) *Standard Zinc Oxide.*

Procedure

10. Carefully weigh out the proportion of the standard extender pigment and standard zinc oxide mutually agreed upon, and rub up to a fairly stiff paste with a glass muller on a glass plate or stone slab with raw linseed oil conforming to the Standard Specifications for Raw Linseed Oil (A.S.T.M. Designation: D 234) of the American Society for Testing Materials.[3] Note the volume of the oil required. Prepare a similar paste with the sample using the same weight of pigment, volume of oil, and number of strokes of the glass muller as used in the preparation of the paste of the standard pigments. Spread the pastes adjacently on a microscope slide, draw a scraper lightly across them so as to present them on an even plane, and judge the color immediately.

Tentative Methods of

ANALYSIS OF DIATOMACEOUS SILICA PIGMENT[1]

A.S.T.M. Designation: D 719 - 43T

ISSUED, 1943.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These methods cover the procedures for the chemical analysis of diatomaceous silica pigment.

LOSS ON IGNITION

Procedure

2. (*a*) Transfer an accurately weighed portion of the sample (about 1 g.) to a previously ignited, weighed porcelain crucible, and ignite at 900 to 1000 C. for 20 min. Cool in a desiccator, and weigh. Heat again for 5 min. to check the loss in weight.

(*b*) *Calculation.*—Calculate the percentage loss on ignition as follows:

$$\text{Loss on ignition, per cent} = \frac{\text{wt. decrease of sample after ignition}}{\text{wt. of sample}} \times 100$$

MATTER SOLUBLE IN HYDROCHLORIC ACID

Procedure

3. (*a*) Transfer 2.5 g. of the sample to a 250-ml. volumetric flask, add 25 ml. of HCl (1:2), and boil for 5 min. Cool, dilute to 250 ml. with water, shake thoroughly, and allow to settle. Decant the solution through a dry filter, discarding the first 20 ml., and catch 100 ml. of the subsequent filtrate in a volumetric flask. Transfer the solution to a tared porcelain dish, and evaporate to dryness.

(*b*) *Calculation.*—Calculate the percentage of matter soluble in HCl as follows:

$$\text{Matter soluble in HCl, per cent} = \frac{\text{wt. of residue}}{\text{wt. of sample}} \times 100$$

MOISTURE AND OTHER VOLATILE MATTER

Procedure

4. (*a*) Transfer 1 g. of the sample to a wide-mouth, short weighing bottle provided with a glass stopper. Heat, with the stopper removed, for 2 hr. at 105 ± 2 C., insert the stopper, cool, and weigh.

(*b*) *Calculation.*—Calculate the percentage moisture and other volatile matter as follows:

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Accepted by the Society at annual meeting, June, 1943.

Moisture and other volatile matter, per cent =

$$\frac{\text{wt. loss on heating}}{\text{wt. of sample}} \times 100$$

VOLUME OF SETTLING IN PETROLEUM SPIRITS

Procedure

5. Transfer 3 g. of the sample to a 100-ml. graduated cylinder. Add mineral spirits, conforming to the Standard Specifications for Petroleum Spirits (Mineral Spirits) (A.S.T.M. Designation: D 235) of the American Society for Testing Materials,[3] until a total volume of 100 ml. is obtained. Disperse the mixture by inverting the cylinder 50 times, and then allow to stand for 1 hr. Read the volume of the settled pigment.

COARSE PARTICLES

(*Total Residue Retained on No. 325 (44-micron) Sieve*)

Apparatus

6. *Sieve.*—A No. 325 (44-micron) sieve conforming to the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials[3] shall be used.

Procedure

7. (*a*) Dry a No. 325 (44-micron) sieve in an oven at 105 to 110 C., cool, and weigh accurately. Transfer 10 g. of the sample to a mortar, moisten with alcohol, add 100 ml. of water, thoroughly mix by gentle pressure with the finger to break up all lumps, and wash through the sieve with a gentle stream of water, brushing gently with a camel's hair brush until nothing more passes through the sieve. Dry the sieve for 1 hr. at 105 to 110 C., cool, and weigh.

(*b*) *Calculation.*—Calculate the percentage of coarse particles as follows:

Coarse particles, per cent =

$$\frac{\text{wt. increase of sieve}}{\text{wt. of sample}} \times 100$$

COLOR

Standard Pigment

8. (*a*) *Standard Extender Pigment.*
(*b*) *Standard Zinc Oxide.*

Procedure

9. Carefully weigh out the proportions of the standard extender pigment and standard zinc oxide mutually agreed upon, and rub up to a fairly stiff paste with a glass muller on a glass plate or stone slab with raw linseed oil conforming to the Standard Specifications for Raw Linseed Oil (A.S.T.M. Designation: D 234) of the American Society for Testing Materials.[3] Note the volume of the oil required. Prepare a similar paste with the sample, using the same weight of pigment, volume of oil, and number of strokes of the glass muller as used in the preparation of the paste of the standard pigments. Spread the pastes adjacently on a microscope slide, draw a scraper lightly across them so as to present them on an even plane, and judge the color immediately.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Tentative Specifications for

OITICICA OIL (PERMANENTLY LIQUID)[1]

A.S.T.M. Designation: D 601 – 41 T

ISSUED, 1941.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These specifications cover oiticica oil that has been treated to render it permanently liquid.

Properties

2. Oiticica oil (permanently liquid) shall conform to the following requirements:

Specific gravity, 15.5/15.5 C., min.	0.978
Viscosity, min., poises	18
Acid number, max.	8.0
Iodine number (Wijs), min.	135
Color (Gardner 1933 Standards),[a] max.	11
Heating test, max., minutes	17
Refractive index at 25 C., min.	1.5100
Matter insoluble in chloroform, max., per cent	0.1

[a] H. A. Gardner, "The Physical and Chemical Examination of Paints, Varnishes, Lacquers, and Colors," Ninth Edition, p. 69.

Methods of Testing

3. (*a*) The properties enumerated in these specifications, with the exception of viscosity and color, shall be determined in accordance with the Standard Methods of Testing Drying Oils (A.S.T.M. Designation: D 555) of the American Society for Testing Materials.[3]

(*b*) Viscosity shall be determined in accordance with the Standard Methods of Testing Oleoresinous Varnishes (A.S.T.M. Designation: D 154) of the American Society for Testing Materials.[3]

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Accepted by the Society at annual meeting, June, 1941.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Tentative Methods of

SAMPLING AND TESTING TURPENTINE[1]

A.S.T.M. Designation: D 233 - 44 T

ISSUED, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These methods cover procedures for sampling and testing turpentine and pinene.

SAMPLING

Sampling

2. (*a*) The method of sampling specified in Paragraph (*b*) or (*c*) shall be used, according to the special conditions that obtain.

(*b*) *From Loaded Tank Car or Other Large Vessel.*—The composite sample taken shall be not less than ½ gal. and should consist of small samples of not more than 1 qt. each, taken from near the top and bottom by means of a metal or glass container with removable stopper or top.[3] This device, attached to a suitable pole, shall be lowered to the desired depth, when the stopper or top shall be removed and the container allowed to fill. If a sample from close to the bottom of a tank shows a decided difference in color or appearance from samples taken at other depths, an extra bottom sample shall be taken and examined separately from the composite sample. In such case the composite sample shall not include any portion of such bottom sample.

(*c*) *From Barrels and Drums.*—At least 5 per cent of the packages in any shipment shall be represented in the sample. The purchaser may increase the percentage of packages to be sampled at his discretion, and it is recommended that every package be sampled in the case of expensive terpene hydrocarbons that are bought in small quantity. A portion shall be withdrawn from about the center of each package sampled by means of a "thief" or other sampling device. The composite sample thus obtained shall be not less than 1 qt. and shall consist of equal portions of not less than ½ pt. from each package sampled.

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[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Accepted by Committee E-10 on Standards, October 30, 1944.

Prior to their present publication as tentative, these methods were published as standard from 1926 to 1944, being revised in 1933 and 1936. Prior to adoption in 1926, these methods were published as part of the Standard Specifications for Turpentine (D 13 - 24).

[3] Detailed description of equipment suitable for such sampling is given in the Standard Methods of Sampling Petroleum and Petroleum Products (A.S.T.M. Designation: D 270), 1944 Book of A.S.T.M. Standards, Part III.

DETECTION AND REMOVAL OF SEPARATED WATER

Procedure

3. Draw a portion by means of a glass or metal container with a removable stopper or top,[3] or with a "thief," from the lowest part of the container, or by opening the bottom valve of the perfectly level tank car. If water is found to be present, draw it all out, record the quantity, and deduct it from the total volume of liquid delivered.

APPEARANCE

Procedure

4. Examine a portion of the sample after agitation to determine whether its appearance conforms to the specifications.

COLOR

Apparatus

5. *Colorimeter Tube.*—The colorimeter tube shall be a straight, cylindrical tube of clear, colorless, soda-lime glass. It shall be 230 to 280 mm. in length, 21 ± 1 mm. in inside diameter, and 1¼ to 1½ mm. in wall thickness. The bottom may be (*1*) blown with the tube, of a uniform thickness of 1.0 to 1.5 mm., with the outer surface flat-ground and polished, and showing little or no distortion of the field of view when the tube is filled to a depth of 50 mm. with the sample; or it may be (*2*) a separate colorless glass plate vitreously fused or sealed to the bottom of the cylinder, 1.0 to 1.5 mm. in thickness, with the outer surface flat-ground and polished, and having a diameter not more than 1 mm. greater than the outside diameter of the cylinder. Graduation lines shall be ruled or etched on the outside of the cylinder at each 2 mm. from the level of the inner surface of the bottom to a height of 200 mm., each tenth line being longer and marked to show the distance from the bottom.

Procedure

6. (*a*) Fill one colorimeter tube to a depth of from 40 to 50 mm. with the sample and place over or under it (in a colorimeter if available) a No. 2.0 yellow Lovibond glass. Place over or under a second colorimeter tube a No. 1.0 yellow Lovibond glass, and then pour the sample into this tube until the colors of the fields of vision or beams of light passing through the tubes and the superimposed glasses are as nearly alike as possible. If the difference in the depths of the columns of sample is 150 mm. or more, the material is "Water-white"; if this difference is less than 150 mm. but not less than 50 mm., the material is "Standard."

(*b*) *Alternative Procedure A.*—Compare the color of the sample in colorimeter tubes or in any other suitable apparatus (such as ordinary 4-oz. oil sample bottles having flat polished bottoms), with the color of an equal depth (at least 100 mm.) of freshly prepared solutions containing the following amounts of $K_2Cr_2O_7$ and $CoCl_2$ per liter (Note 1):

Test Solution	$K_2Cr_2O_7$	$CoCl_2 \cdot 6H_2O$
For Water-white.....	0.0072 g.	0.07 g.
For Standard.....	0.0180 g.	0.15 g.

NOTE 1.—These aqueous standard solutions may be conveniently made up by diluting stock solutions, as follows:

Solution A.—Dissolve 1.80 g. of c.p. $K_2Cr_2O_7$ in 1 liter of distilled water containing 1 ml. of HCl (sp. gr. 1.18).

Solution B.—Dissolve 1.00 g. of $CoCl_2 \cdot 6H_2O$ in 1 liter of distilled water containing 20 ml. of HCl (sp. gr. 1.18).

"Water-white" Test Solution.—Add 2.0 ml. of Solution A to 35 ml. of Solution B and dilute to 500 ml. with distilled water.

"Standard" Test Solution.—Add 5.0 ml. of Solution A to 75 ml. of Solution B and dilute to 500 ml. with distilled water.

(*c*) *Alternative Procedure B.*—Compare the color of the sample, in the same manner as described in Paragraph (*b*), with solutions of K_2PtCl_6 (Note 2). (Do not use $CoCl_2$ with this reagent for this test.)

NOTE 2.—These aqueous standard solutions may be conveniently made up by diluting a stock solution, as follows:

Stock Solution.—Dissolve 0.6225 g. of c.p. K_2PtCl_6 in distilled water, add 50 ml. of c.p. HCl (sp. gr. 1.18), and dilute to 500 ml.

"Water-white" Test Solution.—Dilute 22.0 ml. of the stock solution to 250 ml.

"Standard" Test Solution.—Dilute 47.5 ml. of the stock solution to 250 ml.

These diluted solutions are relatively stable, for at least six months, and may be preserved for recurrent use by sealing the bottles with clean, new, tightly fitting cork stoppers that have been impregnated and waterproofed by immersion in hot melted paraffin wax for 30 min.

(*d*) *Alternative Procedure C.*—Determine the color with a Saybolt Chromometer according to the method given in the Standard Method of Test for Color of Refined Petroleum Oil by Means of Saybolt Chromometer (A.S.T.M. Designation: D 156).[4] If the color is equal to Saybolt No. 19 or above, the material is "Water-white." If the color is darker than Saybolt No. 19 but not darker than Saybolt No. 10, the material is "Standard."

ODOR

Procedure

7. Compare the odor of the sample with an agreed-upon water-free reference sample kept in the dark in a completely filled, well-stoppered bottle. In the absence of such a sample, compare with samples of known purity similarly preserved.

SPECIFIC GRAVITY

Procedure

8. Determine the specific gravity at 15.5/15.5 C. by any convenient method having a precision of 0.0005. Determinations made at any other temperature using apparatus standardized at 15.5 C. shall be corrected by adding to or subtracting from the observed reading 0.00082 for each degree Centigrade that the temperature of the liquid is above or below 15.5 C. If the determination is made with apparatus calibrated for a temperature other than 15.5 C., the observed reading shall first be calculated to density at the temperature of observation, then converted to density at 15.5 C. by applying the above factor, and finally converted to specific gravity by dividing the calculated density by 0.999 (the density of water at 15.5 C.).

REFRACTIVE INDEX

Procedure

9. Determine the refractive index with an accurate instrument, at 20 C. if possible. If determined at any other temperature, correct the reading obtained to 20 C. by adding or subtracting 0.00045 for each degree Centigrade that the temperature at which the determination was made is, respectively, above or below 20 C.

DISTILLATION

Apparatus

10. Use the type of distillation apparatus described in the Standard Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (A.S.T.M. Designation: D 86),[4] with the following exceptions:

(*1*) For testing turpentine or pinene use an A.S.T.M. Turpentine Distillation Thermometer, 76-mm. (3-in.) partial immersion, having a range of 147 to 182 C., and conforming to the requirements for thermometer E 1 (27C – 42) as prescribed in the Standard Specifications for A.S.T.M. Thermometers

[4] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(A.S.T.M. Designation: E 1 – 44);[4] or an A.S.T.M. Solvents Distillation Thermometer, 100-mm. (3.94-in.) partial immersion, having a range of 95 to 255 C., and conforming to the requirements for thermometer E 1 (42C – 44T) as prescribed in the Tentative Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1 – 44 T).[4]

(*2*) Ice is not necessary in the condenser bath. It is permissible to use a glass Liebig condenser 22 in. in length with 16 in. in contact with the cooling water and fitted with an adapter to extend 1 in. into the receiving graduate.

Procedure

11. (*a*) Using the receiving graduate, transfer exactly 100 ml. of the sample directly into the flask, allowing none to run into the side tube. If the sample contains dissolved or suspended water it is advisable to add a few small pieces of pumice or broken glass to promote smooth distillation. Insert the thermometer, so placed that the top of the mercury bulb (or the top of the contraction chamber if the Solvents Distillation Thermometer is used) is level with the bottom of the side tube. Connect the side tube to the condenser, with the bottom of the flask resting securely in the opening in the asbestos board. Apply heat cautiously, and regulate it so that the first drop of condensate falls from the condenser in not less than 5 nor more than 10 min. Record as the *initial boiling point* the thermometer reading when the first drop falls from the end of the condenser, correcting for barometric pressure as described in Section 12. When the distillation begins, regulate the heat so that the distillate is collected at a rate of not less than 4 nor more than 5 ml. per min. (approximately 2 drops per sec.). Discontinue the distillation when the temperature reaches that specified for the minimum percentage requirement (usually 170 C. for turpentine), after correcting for barometric pressure. Allow the condenser to drain and read the percentage distilled.

(*b*) The percentages distilled below successive selected temperatures and the temperature at which each successive 10 ml. distills may also be determined, if desired, making the necessary correction of the temperature for barometric pressure.

TABLE I.—TEMPERATURE CORRECTION (DEGREES CENTIGRADE) FOR BAROMETRIC PRESSURE.[a]

Observed Barometric Pressure, mm.	Aneroid Barometer	Mercurial Barometer			
		Temperature of Barometer			
		20 C.	25 C.	30 C.	35 C.
780.....	−1.14	−1.00	−0.96	−0.92	−0.88
770.....	−0.57	−0.43	−0.39	−0.36	−0.32
760.....	0.00	+0.14	+0.18	+0.21	+0.25
750.....	+0.57	+0.71	+0.74	+0.78	+0.82
740.....	+1.14	+1.26	+1.30	+1.34	+1.38
730.....	+1.71	+1.84	+1.87	+1.91	+1.95
720.....	+2.28	+2.41	+2.45	+2.48	+2.52
710.....	+2.85	+2.98	+3.01	+3.05	+3.09
700.....	+3.42	+3.55	+3.58	+3.61	+3.65

[a] These corrections are calculated as follows: The observed barometric pressure is first corrected to what it would be at 0 C., by means of the table in *Circular F*, Instrument Division, U. S. Weather Bureau. The corrected barometric pressure is then subtracted from 760 (or *vice versa*) and the difference is multiplied by 0.057 C. to give the temperature correction shown in Table I. (According to Landolt-Bornstein "*Physikalish-Chemische Tabellen*," Fourth Edition, Table 127, p. 436, the distillation temperature of turpentine is affected plus or minus 0.057 C. for each millimeter variation of the barometer above or below the normal 760 mm. at 0 C.).

Barometer Correction

12. (*a*) The distilling temperature of turpentine (and pinene) is affected by 0.057 C. for each millimeter variation of the atmospheric pressure. Therefore, the distillation temperatures observed or specified shall be corrected to permit the distillation to be conducted as though the barometer reading, corrected to 0 C., were exactly 760 mm.

(*b*) When about to begin the distillation, observe and record the barometric pressure and the temperature of the barometer. (No temperature correction is necessary for aneroid type barometers.) From Table I determine the proper

temperature correction corresponding to these atmospheric conditions, interpolating to the nearest 0.1 C. If the barometric pressure, corrected to 0 C., is below 760 mm., the temperature correction must be added to the initial boiling point and subtracted from the end-point temperature; if above 760 mm., the correction must be subtracted from the initial boiling point and added to the end-point temperature.

NOTE: *Example.*—Suppose the observed barometric pressure is 750 mm. at 32 C., and the initial boiling point is observed to be 155.2 C. From Table I the temperature correction is seen to be 0.8 C. Therefore, the corrected boiling point is 155.2 + 0.8 = 156 C. Furthermore, the temperature observation point at the end of distillation (170 C. at 760 mm.) must be altered to the same extent. Since the turpentine is distilling 0.8 C. below what it would at normal pressure, distillation must be discontinued at 169.2 C. to determine the percentage distilling below 170 C. at 760 mm. pressure.

POLYMERIZATION

Reagent

13. (*a*) *Sulfuric Acid (38 N).*—In a weighed, glass-stoppered bottle (the usual 2½-liter acid bottle is convenient) mix H_2SO_4 (sp. gr. 1.84) with fuming sulfuric acid (Note). If the concentration of the fuming acid is designated by its content of free SO_3, then the following proportions by weight will ordinarily yield a mixture slightly stronger than 38 *N*:

Free SO_3 in Fuming Sulfuric Acid, per cent	Parts by Weight of Fuming Sulfuric Acid	Parts by Weight of H_2SO_4 (sp.gr. 1.84)
50	100	150
20 to 30....	100	75
15 to 20....	100	50

Determine the exact strength of the mixed acid in terms of H_2SO_4 by titrating a diluted aliquot with standard alkali, observing the usual precautions necessary for the accurate analysis of fuming acids. In the same way determine the percentage of H_2SO_4 in the stock of concentrated acid (sp. gr. 1.84). From these data calculate the quantity of H_2SO_4 (sp. gr. 1.84) that must be added to the mixed acid to bring it to a concentration of 100.92 per cent of H_2SO_4.

NOTE.—Fuming acid containing oxides of nitrogen to prevent freezing should not be used.

(*b*) After adjusting the concentration by adding the required quantity of H_2SO_4 (sp. gr. 1.84), thoroughly shake the bottle of mixed acid and again determine its concentration. The permissible variation is plus or minus 0.15 per cent of H_2SO_4. As a final check it is advisable to run a polymerization test on a sample of turpentine of known purity. The residue should not exceed 2 per cent.

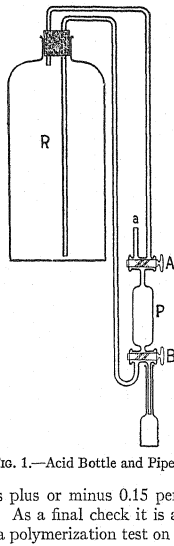


FIG. 1.—Acid Bottle and Pipette.

(*c*) Special precautions shall be taken to prevent dilution of the 38 *N* H_2SO_4 by the absorption of atmospheric moisture. The arrangement shown in Fig. 1 is most suitable for storing and delivering measured quantities of this reagent. With the three-way stopcocks *A* and *B* in the

position shown, acid is siphoned into the pipette *P*, the displaced air passing into *R*. To empty the pipette, *A* and *B* are turned to the position shown by the broken lines, air passing in at *a*. The acid adhering to the walls of the pipette dries this air so that when it passes into *R* on again filling the pipette there is no accumulation of moisture in the acid remaining in the reservoir. If such an arrangement is not to be had, the acid should be kept in bottles of not more than 500-ml. capacity having tight-fitting glass stoppers.

Procedure

14. Place 20 ml. of 38 *N* H_2SO_4 in a graduated, narrow-neck Babcock bottle, stopper, and place in ice water to cool. Add slowly from a pipette, a little at a time, while shaking gently after each addition, 5 ml. of the material to be examined. Add the turpentine drop by drop, with sufficiently vigorous shaking after each addition to mix the sample intimately into the acid. Turpentine is lighter than and not readily miscible with the acid; hence the shaking shall be performed in such a way as to mix the oil into the acid. Continue the shaking after all of the sample has been added, keeping the temperature down to 60 to 65 C. by continued immersion in the ice water. When the mixture no longer warms on shaking, agitate thoroughly, and place the bottle in a water bath and heat at 60 to 65 C. for not less than 10 min., keeping the contents of the bottle thoroughly mixed by vigorous shaking for 30 sec. each time, six times during the period. Do not stopper the bottle after the sample has been added, as it may explode. Cool to room temperature, fill the bottle with H_2SO_4 (sp. gr. 1.84) until the unpolymerized oil rises into the graduated neck, and centrifuge from 4 to 5 min. at not less than 1200 rpm., or for 15 min. at 900 rpm., or allow to stand, lightly stoppered, for 12 hr. Calculate the percentage, note the consistency and color, and determine the refractive index (at 20 C.) of the unpolymerized residue.

Tentative Specifications for

ORANGE SHELLAC AND OTHER LACS[1]

A.S.T.M. Designation: D 237 – 43 T

ISSUED, 1943.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These specifications cover six grades of orange shellac and other lacs designated grades A, B, C, D, E, and F. Stick-lac and seed-lac are not included under these grades.

Properties

2. The material shall conform to the requirements prescribed in Table I.

NOTE 1.—Grades A, B, and C should show no presence of rosin when tested in accordance with the qualitative test described in Section 15 of the Standard Methods of Sampling and Analysis of Shellac (A.S.T.M. Designation: D 29).[3]

NOTE 2.—Grade E customarily occurs in the form of circular disks about 3 in. in diameter and $\frac{1}{8}$ in. in thickness and is known to the trade as pure button lac. Grade F is dark garnet in color and is known to the trade as pure garnet lac. Garnet lac also is manufactured in admixture with rosin and as a dewaxed lac, but these are not covered by these specifications.

TABLE I.—REQUIREMENTS FOR ORANGE SHELLAC AND OTHER LACS.

	Grade A	Grade B	Grade C	Grade D	Grade E	Grade F
Matter insoluble in specified hot solvents, max., per cent	1.25	2.00	3.00	3.00	1.00	0.50
Iodine number, max	....	....	....	24.3	18.0	18.0
Moisture, max., per cent	2.0	2.0	2.0	2.0	2.0	2.0
Wax, max., per cent	5.5	5.5	5.5	5.5	4.0	3.5
Orpiment, max. per cent	0.025	0.025	0.20	0.20	none	none
Matter soluble in water, max., per cent	0.5	0.5	0.5	0.5	0.5	0.5
Ash, max., per cent	1.0	1.0	1.0	1.0	0.5	0.5
Color	when specified, the color shall be no darker than that of a sample agreed upon by the seller and the purchaser.[a]					

[a] Attention is called to the fact that the seller and the purchaser must agree upon one of the two methods for determining color appearing in A.S.T.M. Methods D 29.[3]

Methods of Testing

3. The material shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the Standard Methods of Sampling and Analysis of Shellac (A.S.T.M. Designation: D 29).[3]

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Accepted by Committee E-10 on Standards, August 30, 1943.

Prior to their present publication as tentative, these specifications were published as tentative from 1926 to 1927. They were adopted in 1927, published as standard from 1927 to 1930, but withdrawn and replaced in 1930 by D 237 – 29 T which was published as tentative from 1929 to 1933, being revised in 1933. They were again adopted as standard in 1933, published as standard from 1933 to 1943, being revised in 1941, but withdrawn, revised and republished as tentative in 1943.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Tentative Method of Test for

COLOR OF ORANGE SHELLAC[1]

A.S.T.M. Designation: D 29 – 41 T

ISSUED, 1941.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. This method of test is intended for the determination of the color of orange shellac when it is desired that the results be expressed in terms of numerical values.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Funnel.*—A No. 1 Büchner funnel of Coors porcelain, 5.6 cm. in inside diameter.

(*b*) *Colorimetric Tubes.*—Nessler color comparison tubes, approximately 20 mm. in diameter and 30 cm. in length.

(*c*) *Standard Light Source.*—A light source consisting of a 100-w. Mazda, nitrogen filled, frosted glass, daylight lamp mounted at the center of the back wall of a 10-in. (25.4-cm.) cubic box so that the tip of the bulb faces front. The front opening of the box shall be covered with a white opaque paper or a ground-glass plate. The lamp shall be inserted in a socket which just protrudes through the wall of the box so that the distance from the tip of the lamp to the paper or glass is approximately 4.5 in. (11.4 cm.). The five inside walls of the box shall be painted white. Any other suitable apparatus or standard daylight lamp may be used, provided it produces light of the same characteristics as specified above.

Reagents

3. (*a*) *95 Per Cent Alcohol.*—Specially denatured 190 proof alcohol; either formula No. 1 or formula No. 30 of the U. S. Internal Revenue Bureau.

(*b*) *Filter Aid.*—Any high-grade analytical filter cel for rapid flow.

(*c*) *Ferric Sulfate.*—Any hydrated c.p. analyzed reagent.

(*d*) *Standard Sodium Hydroxide Solution.*—Dissolve 40 g. of pure NaOH in 500 ml. of distilled water and dilute to 1 liter in a volumetric flask. Standardize against the correct weight of pure oxalic acid dihydrate. The solution should have a strength of 1 *N*.

(*e*) *Standard Sodium Thiosulfate Solution.*—Dissolve pure sodium thiosulfate

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] This method is in effect a tentative revision of, and is intended to be added, when adopted, to the present Standard Methods of Sampling and Analysis of Shellac (D 29 – 40), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

in distilled water that has been previously boiled to free it from carbon dioxide, in such proportion that 49.66 g. of crystallized sodium thiosulfate will be present in 1 liter of the solution. It is best to let this solution stand about 2 weeks before standardizing. Standardize with pure resublimed iodine or potassium bi-iodate. This solution will be approximately 0.2 *N*. Preserve in a brown stock bottle with a guard tube filled with soda lime.

(*f*) *Starch Solution.*—Make a paste of 0.2 g. of soluble starch (potato starch) in cold water and pour into 100 ml. of boiling water, cool, and bottle.

(*g*) *Potassium Iodide.*—Iodate-free crystal.

(*h*) *Nickel Sulfate.*—Analytical reagent grade, $NiSO_4 \cdot 6H_2O$.

Preparation of Color Standards

4. (*a*) *Stock Solutions:*

Ferric Sulfate.—Dissolve 400 g. of $Fe_2(SO_4)_3 \cdot XH_2O$ (c.p. analyzed reagent grade) in about 600 ml. of water by heating to boiling while stirring constantly. After complete solution, cool to room temperature and dilute to 1 liter in a volumetric flask. Standardize this stock solution by titrating with iodine in the following manner: Dissolve 20 g. of potassium iodide in 30 ml. of water and add 5 ml. of the stock solution. Add to this approximately 2 per cent by weight of H_2SO_4 (sp. gr. 1.84). Allow to stand for 5 min. and titrate with 0.2 *N* sodium thiosulfate in the usual manner, not adding the starch until near the end of the titration. Run at the same time a blank determination of the potassium iodide solution and correct for any nonuniformity of this reagent. Adjust the stock solution to approximately 0.725 *M* $Fe_2(SO_4)_3$ (Note); 5 ml. of the stock solution should then be equivalent to 36.2 ± 0.8 ml[3] of 0.2 *N* sodium thiosulfate solution.

NOTE.—The amount of coordinated water in ferric sulfate may vary, and has to be taken into account in weighing out the required amount of ferric salt. It is usually about 6 mols per mol of ferric sulfate for the powdered analyzed reagent.

Nickel Sulfate.—Dissolve 50 g. of $NiSO_4 \cdot 6H_2O$ in about 300 ml. of water. After complete solution, dilute to 500 ml. in a volumetric flask.

(*b*) *Reference Standard for All Grades.*[3] —Transfer exactly 20 ml. of the stock ferric sulfate solution (Paragraph (*a*)) to a 100-ml. volumetric flask. Add 11.5 ml. of the 1.0 *N* aqueous NaOH solution from a burette, and shake until all the precipitate formed has dissolved. Then, add 10 ml. of the stock nickel sulfate solution and dilute to 100 ml. with distilled water. If any of the solutions show permanent precipitates, prepare fresh stock solution for this purpose.

Procedure

5. (*a*) Transfer exactly 5 g. of the sample to a clean 4-oz. wide-mouth bottle and add exactly 50 ml. of alcohol. Shake until the solution is complete and then cool to 10 C. Place a 5.5-cm. Whatman No. 5 filter paper or its equivalent in the Büchner funnel which has been mounted in the neck of a 2-l. suction flask with a rubber stopper. Pour evenly upon the filter paper a 1-g. suspension of filter aid in 50 ml. of alcohol, and suck completely dry with a partial vacuum, using a water suction pump. Remove the funnel, and add about 400 ml. of alcohol to the flask. Place an 8-in. test tube in the suction flask, supporting, if necessary, with filter paper, so that the tip of the Büchner funnel when inserted in the neck of the flask will come well within the open

[3] This standard color solution was designed to match the No. 5 iodine color standard of the Angelo color method which has been used by various laboratories for obtaining numerical color values for lacs. It is closer in hue to most lacs than the iodine solution and hence easier to match.

test tube. Reinsert the funnel in the flask.

(*b*) Add 1 g. of filter aid to the cold varnish and stir thoroughly. Transfer completely to the Büchner funnel and filter at the rate of 2 drops per second by means of a carefully regulated vacuum. This may be conveniently done by the use of a water pump to which has been attached a trap carrying a stopcock to admit air. Slowly increase the amount of vacuum toward the end of the filtration in order to maintain a constant filtration rate until the filtration is practically complete, and then suck dry. The final volume in the test tube should be 49 to 50 ml. If it is less than 48 ml., repeat the procedure. (Note 1.)

NOTE 1.—The method used in preparing the clear varnishes for comparison is very important. A slight loss in alcohol will materially affect the color of varnish when diluted for comparison. Alcohol is placed in the flask to avoid undue evaporation of the solution during filtration. The rate of filtration is an important factor.

(*c*) Transfer 10 ml. of filtered solution to one of the colorimetric tubes, and compare with 10 ml. of the standard color solution by viewing the tubes transversely in front of the standard light source. Dilute the shellac solution with alcohol until it matches the reference standard color. Report the volume in milliliters of the diluted solution; this is taken as the color number of the sample. The color of the filtered solution shall be determined the same day upon which the samples are dissolved. (Note 2.)

NOTE 2.—Most filtered solutions made by dissolving shellac that has been ground for analysis will darken appreciably in color if allowed to stand for periods longer than 12 hr.

Tentative Method of Test for

PHTHALIC ANHYDRIDE CONTENT OF ALKYD RESIN SOLUTIONS[1]

A.S.T.M. Designation: D 563 – 43 T

ISSUED, 1940; REVISED, 1943.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. This method of test covers the procedure for determining the phthalic anhydride content (Note) of alkyd resin solutions.

NOTE.—Other dibasic acids such as maleic also precipitate as potassium salts under the conditions of the test and, if present as esters in the resin would, therefore, be calculated as phthalic anhydride.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Flask and Condenser.*—A 500-ml. soil-digestion flask fitted with an air-cooled glass reflux condenser 32 in. in length. The connection between the flask and condenser shall be a ground-glass joint.

(*b*) *Steam Bath.*

(*c*) *Gooch Crucible.*

(*d*) *Desiccator.*—A desiccator provided with suitable means for evacuation.

Procedure

3. (*a*) Weigh a quantity of the liquid, equivalent to 2 to 3 g. solid content, into the 500-ml. flask. Add 10 ml. of benzol, connect the flask to the condenser, and warm until a homogeneous mixture is obtained; then add 150 ml. of 0.5 *N* KOH in absolute alcohol (Note). Warm on the steam bath at 60 C. for 1 hr. and then reflux gently for 3 hr. Cool, allow to stand for 1 hr., and wash down the sides of the flask with 50 ml. of absolute ether.

(*b*) Filter at room temperature, using a Gooch crucible with an asbestos mat, and wash the precipitate with 50 ml. of a mixture of equal parts of absolute alcohol and ether, using five 10-ml. portions. Do not allow air to suck through the crystals, as they are strongly hygroscopic and may absorb moisture from the air. Dry for 10 min. in an oven heated to 60 C., and then to constant weight over sulfuric acid in an evacuated desiccator. One gram of the precipitate, the potassium alcohol salt of phthalic acids, is equivalent to 0.5139 g. of phthalic anhydride.

NOTE.—The alcohol may be denatured grade 2B, but must be absolute.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Revision accepted by the Society at annual meeting, June, 1943.

Tentative Methods of

SAMPLING AND GRADING ROSIN[1]

A.S.T.M. Designation: D 509 - 44 T

ISSUED, 1938; REVISED, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These methods describe the procedures for sampling and determining the grade of rosin delivered in commercial bags, barrels, or drums (Note).

NOTE.—All rosin sold in interstate commerce must be described by reference to the U. S. Standards for rosin, and is therefore subject to grading prior to such sale. The grading procedure described in these methods is used for checking grades or regrading after the rosin has moved from the primary markets to distributing or consuming points.

Apparatus

2. The apparatus (see Fig. 1) for sampling and grading rosin shall consist of the following:

(*a*) *Steel Spike.*—A portable steel spike as shown in *A*, Fig. 1, with removable wooden handle, for obtaining a sample lump. The spike shall have the following approximate dimensions: 30 in. in over-all length, 18½ in. in length for steel shank, and 2 in. in outside diameter at handle end. The total weight of the spike shall be 4½ lb. and the weight of the steel shank 3¼ lb. A suitable spike may also be made by sharpening one end of the drive shaft of an old automobile. The sharpened point shall be off center, and, in order to lessen the chances of breaking or snapping off at the tip, should not be extremely hard.

(*b*) *Sampling Adz.*—A sampling adz as shown in *D*, Fig. 1, specially designed for cutting the "type" or grading sample from the rosin lump. A lather's hatchet with a narrow blade (*C* in Fig. 1) may also be used. The cutting edge shall be kept razor sharp for best results.

(*c*) *Heating Device.*—A heating device having a smooth polished surface for the final smoothing and shaping to size of the cut sample for close grading. It shall be maintained at a temperature just high enough to permit rapid melting of the surface of the rosin but not hot enough to cause the rosin to sizzle and permit scorching. The device shown in *E*, Fig. 1, is a specially designed electrical heater having an angular head of nickel-plated copper, with one fin fastened between electric-heating elements. An ordinary electric flat iron mounted

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-17 on Naval Stores.

[2] Revision accepted by the Society at annual meeting, June, 1944.

in a vertical position, point down, with a rheostat in the circuit, to control the current, also serves the purpose.

(*d*) *Grading Box.*—A comparison or grading box as shown in *B*, Fig. 1, which is not always necessary but may be helpful in grading samples that are doubtful or "close to type," that is, almost but perhaps not quite as good as the standard, or which may be difficult to grade because of some unusual condition such as a reduced brightness or light transmission value caused by cloudiness or the presence of finely divided foreign matter suspended in the rosin. Such a box may be made of $\frac{1}{4}$ in. wood stock, with the following approximate dimensions: 21 in. in overall length at the bottom, $19\frac{3}{4}$ in. in length on top, $4\frac{7}{8}$ in. in width at the wide (eye) end, $2\frac{5}{8}$ in. in width at the small (sample) end of the top, $1\frac{1}{4}$ in. in width of side member, and $1\frac{3}{4}$ in. in overall height (top, bottom, and side). The large end shall be cut out as shown in Fig. 1 to fit the face of the grader while the small end shall be fitted with two flat springs made of $\frac{3}{8}$-in. clock-spring steel, which serve to hold the sample and the standard in place on the extended end or lip of the lower member, in front of two $\frac{1}{2}$-in. square holes cut $\frac{7}{8}$ in. apart, center to center, in a piece of sheet metal forming the small end of the box. A piece of sheet rubber fastened to the lip, acts as a cushion for the samples to rest on. An eye-piece, in the form of a transverse vertical partition having a centrally cut opening or slit $\frac{3}{8}$ in. in width by 3 in. in length, shall be fastened inside the larger end, about 4 in. in from the end. The box shall have an interior finish of dull black, which may be obtained by glueing pieces of black

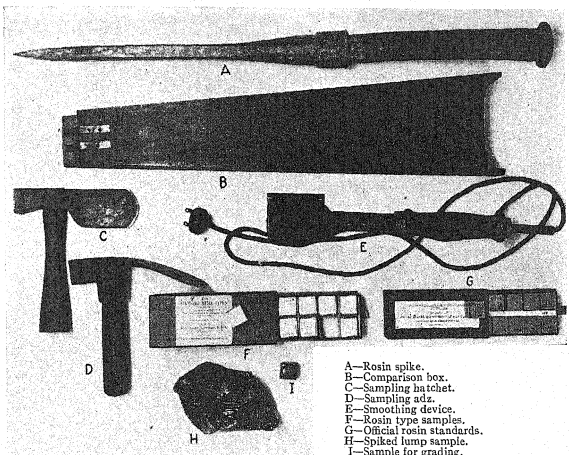


FIG. 1.—Apparatus for Sampling and Grading Rosin.

felt, cut to fit the interior, to the inner side of the several pieces of board from which the box is made.

(*e*) *Official Rosin Standards.*—The Official U. S. Standards for rosin (*G* in Fig. 1) are assembled from glass components and are issued on loan by the U. S. Department of Agriculture,[3] in sets of twelve standards, for the following grades: *X, WW, WG, N, M, K, I, H, G, F, E, D.* There is also a standard for *FF* wood rosin. If "type" samples made of rosin (*F* in Fig. 1), carefully selected and prepared to conform to or match the color of the official permanent government standards, are used, they shall be protected from heat and light when not in use, to prevent deformation and change of color. They shall be tested periodically for color and conformity with the U. S. Standards, and replaced whenever they no longer match the standards.

Sampling

3. (*a*) *Number of Packages to be Sampled.*—A preliminary sampling of 20 per cent of the entire lot or shipment shall be made. If the grade of 85 per cent or more of the number of such preliminary samples agrees with the grade indicated on the package or invoice, with none of the samples disagreeing by more than one grade, the original grading shall be considered confirmed and the shipment accepted as a good delivery. If the grades of more than 15 per cent of the preliminary samples are in disagreement, or if any appreciable number are under grade by more than one grade, additional packages in the lot to make up a total equal to not less than 50 per cent of the entire shipment (preferably the entire shipment if accessible and not too great) shall be sampled. The findings on such larger quantity shall be accepted as the basis for settlement between the purchaser and the seller (Note).

NOTE.—Gum rosin, that is, rosin made from the gum or oleoresin that has exuded from the living tree, is ordinarily made in batches of from 6 to 8 bbl. or drums, weighing from 475 to 600 lb. each. A sample drawn from any one package cannot be expected to represent more than the 6 or 8 bbl. or drums comprising the batch of which the sampled package was a part. The color value of the rosin in any one batch after the rosin has been packaged may show enough variation, due to several factors, to result in a difference in grade (usually not more than one grade) as between different barrels of the batch. The packages comprising a lot of rosin moving or that has moved in commerce from a large concentration and distributing point may represent the production of a number of different producers from widely separated localities, and from gum collected at different seasons of the year, and may also have been graded by a number of different graders. The difficulty of obtaining a correct appraisal of the grading of an entire lot of rosin from a too limited number of samples is thus apparent.

(*b*) *Method of Sampling.*—By means of the spike, the rosin shall be broken into along the lines of a segment of the circular surface, removing the top or surface rosin from one fourth to one third of the surface area, to a depth of several inches. A lump about the size of a clenched fist (*H*, Fig. 1) shall then be broken or pryed loose from such a position that the top or upper side of the lump comes from at least 4 in. below the original surface (2 to 3 in. in the case of drums having permanent heads with small openings). In the case of the usual 100-lb. bags, the sample may come from any convenient location. By means of the sampling adz or hatchet, a grading sample shall be cut or cleaved from the lump as nearly cubical in shape as possible, $\frac{7}{8}$ in. on the side. If desired or indicated as necessary by the results of the cutting, the sample shall be shaped

[3] The standards are obtainable only on security loan from the Naval Stores Section, Cotton and Fiber Branch, War Food Administration, U. S. Dept. of Agric., Washington, D. C., under the Regulations for Enforcement of the Naval Stores Act.

to exact size by quick, intermittent, light applications against the heated smoothing device, removing any melted rosin from the surface with cotton waste or a soft cloth between each application. The final specimen for grading shall be an approximate cube having a thickness of exactly $\frac{7}{8}$ in. (*I*, Fig. 1), between smooth, parallel faces in the direction or dimension through which it is viewed or compared with the grade standard.

Grading

4. (*a*) The sample or "type" shall be compared with the appropriate standard, preferably against an open direct north sky light, or in a direction which excludes direct rays from the sun from passing through the rosin to the eye. The grade of the rosin shall be considered that of the highest or lightest colored standard which the sample equals or excels (is lighter than) in color (Note). Should the color or appearance of the rosin be such that there is a doubt whether the rosin is as light or bright as the standard, the sample and the standard shall be viewed through the grading box or other suitable colorimeter. Then in case of uncertainty the rosin shall be given the benefit of the doubt. For example, if a rosin sample is definitely darker than the *WW* standard, but lighter than the *WG* standard, it is *WG* grade. If, however, the grader is undecided whether the total color of the rosin is "as good as" or equals, or is darker than that of the *WW* standard, the grade is *WW*.

NOTE.—The "color" of a sample of rosin is made up of three attributes: hue, saturation, and brightness. "Hue" alludes to the characteristic described by the words "yellow" or "red." "Saturation" describes the purity or strength of the yellowness or redness of the color. "Brightness" depends on the relative amount of light transmitted by the rosin. The cleanliness of the rosin affects its transparency and therefore its brightness. The terms "lighter than" and its inverse, "darker than" are convenient to describe the difference between a sample and a standard. The rosin may be darker than the standard in one or more of the following ways: (*1*) a redder hue, (*2*) a more saturated hue, and (*3*) a lower brightness.

(*b*) A sample showing two distinct colors usually evidenced by darker streaks through the rosin, indicates a mixture of rosins in the package, in which case the darkest part of the sample or darkest rosin to be found in the package shall determine the grade.

(*c*) A check sample taken from close to the bottom of a barrel or drum shall not be considered representative, because of a natural variation in color sometimes found between the top and bottom rosin, as well as a reduced brightness of the bottom rosin due to a settling of finely divided suspended matter usually present in normal gum rosin. Should such bottom sample show not more than one grade lower than the top sample, the latter shall determine the grade of the rosin in the barrel. If, however, the bottom-head sample is more than one grade lower than the top sample, double filling or "mixed-packing" is indicated, and the bottom-head sample shall determine the grade of the rosin in the package.

(*d*) Rosin which is only slightly cloudy or opaque (caused by occluded moisture or separation of crystals in the solid mass) shall be graded in the usual way if the condition is not such as to prevent an accurate evaluation of the color in comparison with the standards. If, however, the opaqueness is of such a degree that the grade cannot be definitely determined in comparison with the standards, the rosin shall be designated "OPAQUE" and graded "OP," in which case its acceptance and value shall be a matter for settlement between the purchaser and the seller without reference to grade. Such opaque rosin may be

further identified or described as pale, medium, or dark opaque.

Tolerance

5. The tolerance or allowance of 15 per cent variation (Note) described in Section 3 is necessary because careful competent graders rarely get exact duplicate results on all individual samples when regrading a large number of rosin samples. Moreover, a regrade sample, coming from a different position in the package, and after storage and weathering for an indefinite period, may be slightly darker than the original sample on which the grade was based. An allowance for such slight variation must therefore be made.

NOTE.—This tolerance is that recognized by the Federal Government in the administration of the U. S. Naval Stores Act.

Tentative Methods of
SAMPLING AND TESTING DIPENTENE[1]

A.S.T.M. Designation: D 801 – 44 T

ISSUED, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These methods cover procedures for sampling and testing dipentene and related terpene solvents, consisting chiefly of monocyclic terpene hydrocarbons distilling above the range for turpentine.

Sampling

2. Sample the material in accordance with the procedure described in the Standard Methods of Sampling and Testing Lacquer Solvents and Diluents (A.S.T.M. Designation: D 268).[3] If a sample from close to the bottom of a tank shows a decided difference in color or appearance from samples taken at other depths, take an extra bottom sample and examine it separately from the composite sample. In such case the composite sample shall not include any portion of such bottom sample.

Detection and Removal of Separated Water

3. Draw a portion of the dipentene by means of a glass or metal container with a removable stopper or top,[4] or with a thief, from the lowest part of the container, or by opening the bottom valve of the level tank car. If water is found to be present, draw it all out, record the quantity, and deduct it from the total volume of liquid delivered.

Appearance

4. Examine a portion of the sample after agitation to determine whether its appearance conforms to the specifications.

Color

5. (*a*) Compare, in colorimeter tubes or in any other suitable apparatus (such as ordinary 4-oz. oil-sample bottles having flat polished bottoms), the color of the sample with an equal depth of a fresh solution of potassium dichromate in distilled water containing 0.012 g. of $K_2Cr_2O_7$ per liter.

NOTE.—An 150-mm. column of this solution has a color equal to that of a No. 1.5 Lovibond yellow glass.

(*b*) As an alternative method, compare the color of the sample with that of the

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-17 on Naval Stores.

[2] Accepted by the Society at annual meeting, June, 1944.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

[4] Detailed description of equipment suitable for such sampling is given in the Standard Methods of Sampling Petroleum and Petroleum Products (A.S.T.M. Designation: D 270), 1944 Book of A.S.T.M. Standards, Part III.

Saybolt standards as described in the Standard Method of Test for Color of Refined Petroleum Oil by Means of Saybolt Chromometer (A.S.T.M. Designation: D 156).[5]

(*c*) If the color is equal or lighter than the potassium dichromate solution, or if the color is equal to Saybolt No. 16 or above, the dipentene is prime white.

Odor

6. Compare the odor of the sample with an agreed upon water-free reference sample kept in the dark in a completely filled well-stoppered bottle. In the absence of such a reference sample, compare with samples of known purity similarly preserved.

Specific Gravity

7. Determine the specific gravity at 15.5/15.5 C. by any convenient method having a precision of 0.001.

Refractive Index

8. Determine the refractive index with an accurate instrument at 20 C., if possible. If tested at any other temperature, correct the reading obtained to 20 C. by adding or subtracting 0.00045 for each degree Centigrade that the temperature at which the determination was made is, respectively, above or below 20 C.

Distillation

9. (*a*) *Apparatus.*—Use the distillation apparatus described in the Standard Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (A.S.T.M. Designation: D 86),[3] with the following exceptions:

(*1*) *Thermometer.*—Use an A.S.T.M. Partial Immersion Thermometer having a range of −5 to +300 C., conforming to the requirements for this thermometer as prescribed in the Standard Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1),[3] or an A.S.T.M. Solvents Distillation Thermometer having a range of 95 to 255 C. and conforming to the requirements for this thermometer as prescribed in the Tentative Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1).[3]

(*2*) *Condenser.*—A Liebig glass condenser 22 in. in length with 16 in. in contact with the cooling water may be used in place of the bath-type condenser.

(*b*) *Procedure.*—With the receiving graduate, transfer exactly 100 ml. of the sample directly into the flask, allowing none to run into the side tube and allowing the graduate to drain thoroughly. If the sample contains dissolved or suspended water it is advisable to add a few small pieces of pumice or broken glass to promote smooth distillation. Insert the thermometer so that the top of the mercury bulb (or the top of contraction chamber if the Solvents Distillation Thermometer is used) is level with the bottom of the side tube. Connect the side tube to the condenser, with the bottom of the flask resting securely in the opening in the asbestos board. Apply heat cautiously and regulate it so that the first drop of condensate falls from the condenser in not less than 5 nor more than 10 min. Record as the *initial boiling point* the thermometer reading when the first drop falls from the end of the condenser. When the distillation begins, regulate the heat so that the distillate is collected at a rate of not less than 4 nor more than 5 ml. per min. (approximately 2 drops per sec.). Discontinue the distillation when the temperature reaches that specified for the minimum percentage requirement. Allow the condenser to drain and record the percentage distilled.

[5] 1944 Book of A.S.T.M. Standards, Part III.

Polymerization

10. Determine the unpolymerized residue with 38 *N* sulfuric acid in accordance with the procedure described in the Tentative Methods of Sampling and Testing Turpentine (A.S.T.M. Designation: D 233).[3]

Spot Test

11. Transfer 5 drops of the dipentene by means of a small pipette to the center of a clean white filter paper supported on a 7-cm. crystallizing dish and allow the liquid to evaporate at room temperature, away from direct sunlight. After 30 min. observe if there is any oily spot on the filter paper.

Copper Corrosion

12. Place a clean strip of mechanically polished pure sheet copper, about $\frac{1}{2}$ in. in width and 3 in. in length (1.3 by 7.5 cm.) in a glass test tube about $\frac{3}{4}$ in. in width and 18 in. in length (1.9 by 46 cm.). Add sufficient of the sample to be tested to cover the strip completely and heat rapidly to boiling (it is most convenient to heat the tube by immersion in an oil bath maintained at a temperature slightly higher than the initial boiling point of the dipentene). Keep the sample boiling, without any actual distillation taking place, for 30 min., and then examine the copper strip for blackening. Disregard a slight tarnish, but any marked blackening shall be considered cause for rejection.

Flash Point

13. Determine the flash point by means of the Tag Closed Tester as described in the Standard Method of Test for Flash Point by Means of the Tag Closed Tester (A.S.T.M. Designation: D 56).[3]

Moisture

14. Transfer 5 ml. of the sample to a 100-ml. glass-stoppered graduate. Add 60-deg. Baumé gasoline in 5-ml. portions, shaking well after each addition. Both the sample and the graduate shall be at a temperature of 20 C. when used. Add the gasoline in successive additions until the graduate is filled. No sediment or turbidity should develop.

Tentative Methods of
SAMPLING AND TESTING PINE OIL[1]

A.S.T.M. Designation: D 802 - 44 T

ISSUED, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These methods cover procedures for sampling and testing pine oils, and are applicable to both natural pine oils derived from pine stumps either by the steam and solvent process or by destructive distillation, and also to synthetic pine oils obtained by the chemical hydration of terpene hydrocarbons.

Sampling

2. Sample the material in accordance with the procedure described in the Standard Methods of Sampling and Testing Lacquer Solvents and Diluents (A.S.T.M. Designation: D 268).[3]

Appearance

3. Examine a portion of the sample to determine its clarity and freedom from foreign matter and separated water.

Color

4. Compare the color of the sample in any suitable or designated apparatus with the accepted or specified color standard.

Specific Gravity

5. Determine the specific gravity at 15.5/15.5 C. by any convenient method having a precision of 0.0005. Determinations made at any other temperature using apparatus standardized at 15.5 C. shall be corrected by adding to or subtracting from the observed reading 0.0008 for each degree Centigrade that the temperature of the liquid is above or below 15.5 C.

Refractive Index

6. Determine the refractive index with an accurate instrument at 20 C., if possible. If tested at any other temperature, correct the reading obtained to 20 C. by adding or subtracting 0.00040 for each degree Centigrade that the temperature at which the determination was made is, respectively, above or below 20 C.

Distillation

7. (*a*) *Apparatus.*—Use the distillation apparatus described in the Standard Method of Test for Distillation of Gaso-

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[2] Accepted by the Society at annual meeting, June, 1944.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

line, Naphtha, Kerosine, and Similar Petroleum Products (A.S.T.M. Designation: D 86),[3] with the following exceptions:

(*1*) *Thermometer.*—Use an A.S.T.M. Partial Immersion Thermometer having a range of − 5 + 300 C., conforming to the requirements for this thermometer as prescribed in the Standard Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1),[3] or an A.S.T.M. Solvents Distillation Thermometer having a range of 95 to 255 C. and conforming to the requirements for this thermometer as prescribed in the Tentative Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1).[3]

(*2*) *Condenser.*—A Liebig glass condenser 22 in. in length with 16 in. in contact with the cooling water may be used in place of the bath-type condenser. The temperature of the cooling water should not be below 20 C.

(*b*) *Procedure.*—With the receiving graduate, transfer exactly 100 ml. of the sample directly into the flask, allowing none to run into the side tube and allowing the graduate to drain thoroughly. If the sample contains dissolved or suspended water it is advisable to add a few small pieces of pumice or broken glass to promote smooth distillation. Insert the thermometer so that the top of the mercury bulb (or the top of the contraction chamber if the Solvents Distillation Thermometer is used) is level with the bottom of the side tube. Connect the side tube to the condenser, with the bottom of the flask resting securely in the opening in the asbestos board. Apply heat cautiously at first. When distillation begins, regulate the heat so that the distillate is collected at the rate of not less than 4 nor more than 5 ml. per min. (approximately 2 drops per sec.).[3] Observe and record the temperature when 5 ml. has collected in the graduated cylinder and again at the 10, 20, 30, 40, 50, 60, 70, 80, 90, and 95-ml. marks (Note).

NOTE.—Because of the frequent presence of a small amount of dissolved water in pine oil, it is unnecessary to attempt to determine the initial boiling point. It is also not customary to determine the end point in the distillation of pine oil.

Polymerization

8. Determine the unpolymerized residue with 38 *N* sulfuric acid in accordance with the procedure described in the Tentative Methods of Sampling and Testing Turpentine (A.S.T.M. Designation: D 233).[3]

Moisture

9. Determine the moisture content in accordance with the Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (A.S.T.M. Designation: D 95),[3] using a glass flask[4] and either petroleum naphtha, toluene, or xylene as the solvent. It is important that the flask be scrupulously clean, for if it shows any white deposits (such as might be caused from attack by alkalies during previous use), the percentage of water may be erroneously high due to a slight decomposition of the terpene alcohols in the pine oil.

Total Terpene Alcohols[5]

10. (*a*) *Apparatus.*—The apparatus shall consist of the following:

(*1*) *Flask.*—A round-bottom, short-neck, flask of 250-ml. capacity.

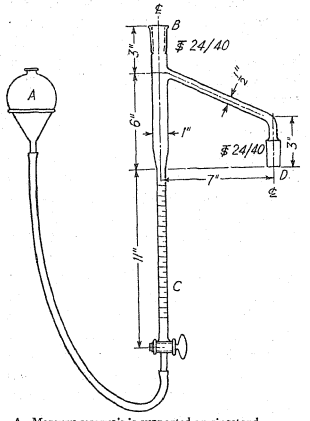
(*2*) *Water Trap.*—A specially constructed glass moisture trap, with dimensions as shown in Fig. 1, with or without stopcock. While not preferred, a Dean and Stark (Barrett type) moisture trap, 20-ml. capacity, may also be used.

[4] Pyrex or Kimble glass is satisfactory for this purpose.

[5] This method of test consists of measuring the water evolved during the catalytic decomposition of the terpene alcohols.

(*3*) *Condenser.*—A straight tube, water-jacketed glass condenser with inside diameter of 10 mm., and a jacket length of 300 mm.

NOTE.—It is preferable to have all joints of the flask, moisture trap, and condenser of the interchangeable ground-glass type, with standard 24/40 taper.



A—Mercury reservoir is supported on ringstand.
B—Connection for 12 in. straight tube water condenser.
C—Receiving trap with capacity of 13 ml., graduated in 0.1 ml.
D—Connection for distilling flask.

FIG. 1.—Apparatus for Determining Alcohols in Pine Oil.

(*4*) *Mercury Reservoir.*—A leveling bulb or globe-shaped separatory funnel, connected to the bottom of the moisture trap with a tightly fitting medium-heavy rubber tube about 24 in. in length. The reservoir rests in a movable ring clamp sliding on a vertical rod.

(*5*) *Heat Source.*—A gas burner or an electric heater having suitable heat control.

(*6*) *Support for Flask.*—Preferably a transite or hard asbestos board with a 2½-in. opening in the center, resting on a ring support. A piece of wire gauze may also be used.

(*b*) *Reagents:*

(*1*) *Kontak Clay* (*Silica Gel*) or finely ground Fuller's earth.

(*2*) *Syrupy Phosphoric Acid* (c.p. 85 per cent).

(*3*) *Dry Dipentene, Xylene, or Petroleum Hydrocarbon Solvent* of similar boiling range, such as mineral spirits.

(*c*) *Procedure.*—Weigh 0.1 g. of Kontak clay or Fuller's earth and brush it into the 250-ml. round-bottom flask, adding a few glass beads. Tare the flask to the nearest 0.1 g. on a triple beam side arm balance, and pipette 100 ± 0.25 g. of the sample into the flask, taking care to avoid dropping any on the neck or outside of the flask. (Should this occur, wipe dry with a cloth moistened with the dipentene or other solvent). From a 1-ml. graduated pipette add 0.3 ml. of phosphoric acid to the flask. Apply a thin film of stopcock lubricant to all ground-glass taper joints, and connect the apparatus. Raise the mercury into the graduated leg of the moisture trap, about 1 in. below the level of the side tube, and fasten the ring support of the reservoir to hold it there. Insert a snug-fitting burette-cleaning brush with long wire handle into the top of the condenser, allowing the brush to remain near the top of the condenser but so that it can be lowered at will down to where the condensation of the distillate takes place. Apply heat gently at first until the reaction gets under way. As the distillation proceeds, and water and dipentene collect in the trap, gradually lower the mercury level in the graduated leg of the trap. Bring down the droplets of water which adhere to the walls of the condenser by adding a few drops of dry solvent at the top of the condenser (enough to wet the

brush) and then moving the brush up and down at the lower end of the condenser. When the reaction appears to have slowed down, as indicated by a slow increase in the volume of trapped water, raise the mercury slowly to return the supernatant liquid to the flask, retaining the water in the trap. Then lower the mercury level to lower the water in the trap and continue the distillation. This raising and lowering of the mercury and the water layer above it, to return the lighter oily portion of the distillate from the trap back into the flask, shall be repeated several times, until it is apparent, from no further increase in the volume of water, that the reaction is complete. The condenser tube shall be entirely free from water droplets at the end of the test.

(*d*) *Calculation.*—From the volume of water collected in the trap deduct the amount of moisture contained in the sample, and calculate the percentage of total alcohols as follows:

$$\text{Total alcohols, per cent} = \text{ml. of } H_2O \times 8.56$$

where:

8.56 = grams of terpene alcohol equivalent to 1 g. of H_2O (Note).

NOTE.—If desired, the moisture may be determined on the total alcohol sample (without addition of a solvent as specified in Section 9), before the addition of the dehydrating agents, using a scrupulously clean flask and the trap shown in Fig. 1.

Tentative Methods of
TESTING TALL OIL[1]

A.S.T.M. Designation: D 803 - 44 T

ISSUED, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These methods cover the test procedures to be applied to whole tall oils or refined tall oils.

Sampling

2. NOTE.—Methods of sampling will be supplied later. Standard Methods of Sampling Petroleum and Petroleum Products (A.S.T.M. Designation: D 270)[3] and the Methods of Sampling Oils and Fats of the American Oil Chemists Society are being studied by the committee. Some of these procedures will probably be proposed for sampling tall oil.

Conditioning

3. Heat the entire sample[4] in a closed container fitted with a capillary vent or its equivalent. Heat by immersion in an open steam or boiling water bath to avoid over heating. Withdraw the specimens only when all crystalline matter has dissolved and when the entire sample becomes a homogeneous fluid after thorough stirring.

PHYSICAL TESTS

VISCOSITY

Procedure

4. Determine the viscosity in accordance with the Standard Method of Test for Viscosity by Means of the Saybolt Viscosimeter (A.S.T.M. Designation: D 88),[5] except that the temperature of testing shall be 210 F. unless a lower temperature is specified but in no case may a temperature of less than 180 F. be used.

POUR POINT

Procedure

5. Determine the pour point in accordance with Section 6 (*b*) of the Standard Method of Test for Cloud and Pour Points (A.S.T.M. Designation: D 97),[3] reporting only the lower pour point.[6]

FLASH AND FIRE POINTS

Procedure

6. Determine the flash and fire points in accordance with the Standard Method

[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-17 on Naval Stores.

[2] Accepted by the Society at annual meeting, June, 1944.

[3] 1944 Book of A.S.T.M. Standards, Part III.

[4] Some kind of agitation, even if done occasionally by hand, saves much time.

[5] Appears in this publication; see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

[6] The upper pour point is not reproducible owing to the tendency of tall oil to crystallize.

of Test for Flash and Fire Points by Means of Open Cup (A.S.T.M. Designation: D 92).[5]

COLOR[7]

Reagent

7. *Benzene, c.p.*

Apparatus

8. The apparatus shall consist of the following:

(*a*) *Light Source.*—A light source of a 100 w. frosted Mazda daylight lamp.

(*b*) *Magnesia Block.*—A magnesia block with dimensions of 1 by 2¾ by 3¾ in.

(*c*) *Tintometer.*—An enclosed Wesson type K60B tintometer consisting of a lightproof box with dull black interior containing the 100 w. frosted Mazda daylight lamp, the block of magnesia with white reflecting surface set at a proper angle to reflect the light vertically upward through the tube containing the sample of oil and through the standard color glasses alongside the tube of oil, and receptacles for holding the tube of oil and the color glasses. An eyepiece with dull black interior finish fits over the oil tube and color glasses so that the light passing through both may be observed simultaneously.

(*d*) *Lovibond Color Glasses.*—Red and yellow color glasses, of suitable numbers to match the color of the oils to be examined. Red glasses shall be standardized by the National Bureau of Standards. Use the correct color value regardless of the numerical value shown on the glasses. The minimum standard set shall consist of the following numbers of red and yellow glasses:

Red.....	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
	1.0	2.0	2.5	3.0	3.5	4.0	5.0	6.0	7.0
	7.6	8.0	9.0	10.0	11.0	12.0	16.0	20.0	
Yellow....	1.0	2.0	3.0	5.0	10.0	15.0	20.0	35.0	50.0 70

Also neutral or colorless glasses.

[7] Adopted from the official methods of the American Oil Chemists' Society.

(*e*) *Color Tubes.*—Color tubes having a flat, smooth polished bottom of clear, colorless glass and of the following dimensions: 154 mm. in over-all length, 19 mm. in inside diameter, 22 mm. in outside diameter, and marked to indicate an oil column of 67 mm.[8]

Procedure

9. (*a*) Fill a tube with the oil to be examined to a depth of 67 mm. This depth is equal to one half the standard column used in oil testing, and is designated half column. Oil shall be at a temperature of 20 to 24 C. and shall be clear and transparent. Filter through a heavy-grade, close-texture, filter paper, if necessary to remove turbidity to permit matching the color (in such cases note on the report that filtering was necessary). If, however, the tall oil under examination is not completely liquid at 24 C., heat slowly with agitation until completely liquefied. Cool the liquefied tall oil to 24 C. and read the color before the sample has recrystallized (Note). Insert the tube containing the tall oil in the tintometer housing and place beside it a tube filled to the 67-mm. mark with distilled water. Place Lovibond red and yellow color glasses over the tube containing the distilled water until a match is obtained, observing the color of the oil and glasses through the eyepiece.

(*b*) In matching the color use the least possible number of glasses to obtain the color match. When matching the color of very light colored tall oils it may be necessary to place three neutral or clear glasses over the tube containing the oil to get a balanced field. Where the color is too dark to read, dilute the tall oil to 5 per cent by weight in benzene[9] and determine its Lovibond color.

[8] This is the standard American Oil Chemist's Society color tube with an additional line marked as specified and is identical with the Standard Color Tube of the American Oil Chemists' Society.

[9] Other solvents may not be used.

Report

10. Report the color as follows:

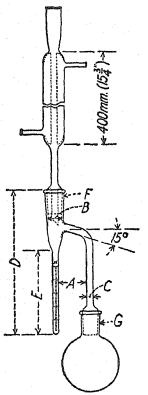
(1) For Half Column of Tall Oil:

Lovibond Half-Column Color..... {Red / Yellow}

(2) For Half Column of 5 per cent Solution of Tall Oil in Benzene:

Lovibond Half-Column Color 5 per cent Solution in Benzene..... {Red / Yellow}

NOTE.—If the sample recrystallizes before a color reading can be obtained, remove the crystals by filtration through a Büchner funnel and determine the color of the filtrate.



A = 45 to 55 mm.
B = 22 to 24 mm., inside diameter.
C = 9 to 11 mm., inside diameter.
D = 235 to 240 mm.
E = 146 to 156 mm.
F and G are interchangeable joints, standard taper 24/40.

FIG. 1.—Assembly of Apparatus for Moisture Test.

MOISTURE

Apparatus

11. The apparatus as shown in Fig. 1 shall consist of the following:

(*a*) *Flask.*—A 500-ml. or a 1-liter short-neck, round-bottom, glass flask, depending upon the weight of the sample used.

(*b*) *Oil Bath.*—An oil bath which does not smoke excessively below 160 C. (Note).

(*c*) *Condenser.*—A reflux condenser connected to the flask and discharging into a trap.

(*d*) *Trap.*—A trap made of well-annealed glass constructed in accordance with Fig. 1 and graduated as shown to contain 5 ml. at 20 C. shall be used. It shall be subdivided into 0.1-ml. divisions, with each 1-ml. line numbered (5 ml. at top). The error in any indicated capacity may not be greater than 0.05 ml.

NOTE.—An oil bath is required because the radiant heat of electric heaters and most gas flames causes sufficient cracking to form appreciable quantities of water especially where impurities such as black liquor, soaps, lignin, or other xylene insoluble matter is present.

Reagent

12. *Xylene, c.p.*

Procedure

13. For tall oils containing up to 1.0 per cent moisture use 500 ± 2 g. of the sample. For tall oil containing 1 per cent or more of moisture use 150 ± 1 g. of the sample. Weigh the tall oil into the flask and add either 200 ml. of xylene for the 500-g. sample or 100 ml. of xylene for the 150-g. sample. Attach the flask to the trap which is connected to the condenser. Prior to starting the determination, fill the receiver with xylene by pouring in through the reflux condenser. So that the refluxing will be under better control, wrap the flask and tube leading to the receiver with asbestos cloth. Heat the oil bath with a gas burner or other source of heat and distill slowly. The rate at the start shall be approximately 100 drops per minute. When the greater part of the water has distilled, increase the distillation rate to 200 drops per minute until no more water is collected.

Purge the reflux condenser during the distillation with 5-ml. portions of xylene to wash down any moisture adhering to the walls of the condenser. The water in the receiver may be made to separate from the xylene by using a spiral copper wire. Move the wire up and down in the condenser occasionally, thus causing the water to settle to the bottom of the receiver. Reflux for at least 2 hr. and shut off the heat at the end of this period. Adjust the temperature of the distillate to 20 C. Read the volume of water.

Calculation

14. Calculate the percentage of moisture from the following formula (Note):

$$\text{Moisture, per cent} = \frac{V}{W} \times 100$$

where:

V = volume of water in milliliters at 20 C., and
W = weight of sample in grams.

NOTE.—Express the results by two digits, for example: 0.31, 1.0, or 3.1 per cent.

ASH

Apparatus

15. *Platinum Dish.*—A platinum dish of 50 to 100-ml. capacity (Note).

NOTE.—A porcelain or silica dish may be used in place of platinum, if the ash is not to be analyzed.

Procedure

16. Heat the platinum dish to redness and, after cooling in a desiccator, weigh to the nearest 0.0001 g. Place an approximately 20-g. sample of tall oil in the dish and weigh to the nearest 0.1 g. (Note 1). Heat the dish gently by means of a bunsen burner until the oil can be ignited at the surface (Note 2). Remove the burner and allow the oil to burn completely. Burn all free carbon on the sides of the dish and heat the residue with a strong flame, or in a muffle furnace, until all carbonaceous matter disappears. After cooling in a desiccator weigh the dish to the nearest 0.0001 g. Repeat the heating until a constant weight is obtained.

NOTE 1.—This size is suitable for ash contents in the range of 0.02 to 0.2 per cent; but in the case of lower or higher ash contents, a larger or smaller sample may be used. In such cases the result shall be specified or reported as follows: "Ash (. . . g. sample) . . . per cent."

NOTE 2.—To avoid foaming and loss of sample in the case of samples containing moisture, it is advisable to add 1 to 2 ml. of absolute alcohol before heating.

Calculation

17. Calculate the percentage of ash from the following formula (Note):

$$\text{Ash, per cent} = \frac{R}{W} \times 100$$

where:

R = weight of residue in grams, and
W = weight of sample in grams, dry basis.

NOTE.—Express the results in two digits.

CHEMICAL ANALYSIS

ACID NUMBER

Reagents

18. (*a*) *Sodium Hydroxide Solution (0.5 M).*—Standardize to plus or minus 0.001 *M*.

(*b*) *Methyl Alcohol.*—Synthetic grade.[10]

(*c*) *Thymol Blue Solution.*—Dissolve 0.1 g. of thymol blue in 20 ml. of hot methanol and dilute to 100 ml. with distilled water.

Procedure

19. In a 250-ml. Erlenmeyer flask[11] of

[10] The pure methanol, 99.5 per cent, sold commercially by most leading producers of methanol is satisfactory. Ethyl alcohol (formula 3A or 30) may be used.

[11] The flasks should be either new or cleaned by rinsing with a hot solution of hydrofluoric acid (2 to 3 per cent). This removes from the flasks the adhering partially disintegrated silicates which would interfere with the determination. This treatment of flasks is not as important for determination of acid number as for determination of saponification number; however, it should be a good precautionary measure.

heat-resistant glass,[12] dissolve 3.95 to 4.05 g. of the sample[13] of tall oil in 100 ml. of methyl alcohol. Twirl the flask to dissolve the sample, using slight heat if necessary. Titrate with standard 0.5 *M* sodium hydroxide solution using 1 ml. of thymol blue solution as an indicator. The end point is a distinct blue (Note).

NOTE.—For dark-colored tall oil the blue color will not be distinct. The end point is taken as the first permanent distinct darkening of the mixture. The end point can be seen more clearly if the titration is stopped just short of the end point and a few drops of thymol blue solution allowed to run down the side of the flask following each addition of 0.1 ml. of standardized NaOH solution.

Calculation

20. Calculate the acid number, expressed in milligrams of KOH per gram, from the following formula:

$$\text{Acid number} = \frac{A \times M \times 56.1}{W}$$

where:

A = milliliters of NaOH solution,
M = molar concentration or normality of the NaOH solution, and
W = weight of sample in grams, dry basis.

SAPONIFICATION NUMBER

Reagents

21. (*a*) *Alcoholic Potassium Hydroxide Solution (0.5 M).*—Dissolve 33.0 g. of pure potassium hydroxide pellets or sticks (85 per cent KOH) in methanol[14] and dilute to 1 liter in a volumetric flask. The solution must be clear. If it contains insoluble matter, it shall be either decanted after settling over night or filtered.

(*b*) *Sulfuric Acid Solution (0.25 M).*—Standardize to plus or minus 0.001 *M*.

(*c*) *Thymol Blue Solution.*—Dissolve 0.1 g. of thymol blue in 20 ml. of hot methanol and dilute to 100 ml. with distilled water.

Procedure

22. (*a*) Weigh 2.95 to 3.05 g. of the sample[13] of tall oil into a 250-ml. Erlenmeyer flask[11] of heat-resistant glass,[12] preferably with ground-glass standard 34/45 taper joint. Add 50.0 ml. of alcoholic potassium hydroxide. Connect the flask to a water condenser. Place the flask in a water bath and allow the solution to reflux for 0.5 hr. Cool and without washing down the condenser titrate immediately with 0.25 *M* sulfuric acid using 1 ml. of thymol blue as indicator.

(*b*) Conduct two or three blank determinations using 50.0 ml. of 0.5 *M* alcoholic KOH.

Calculation

23. Calculate the saponification number, expressed as milligrams of KOH per gram, from the following formula:

$$\text{Saponification number} = \frac{(B - S) \times N \times 56.1}{W}$$

where:

B = milliliters of H_2SO_4 for titration of blank,
S = milliliters of H_2SO_4 for titration of sample,
N = normality of the H_2SO_4 solution, and
W = weight of sample in grams, dry basis.

ROSIN ACIDS NUMBER

Modified Wolff Method

Reagents

24. (*a*) *Methanol.*—Pure methanol (99.5 per cent) sold commercially by most of the leading producers of methanol.

(*b*) *Methyl Sulfuric Acid Solution.*—Slowly pour 100 g. of sulfuric acid (sp. gr.

[12] Pyrex or Kimble glass is very satisfactory for this purpose.
[13] It is desirable to stay within rather narrow limits as to size of sample, in order not to introduce too large variations in the relative concentrations of tall oil, alcohol, and alkali solutions.
[14] Ethyl alcohol (formula 3A or 30) may be used.

1.82 to 1.84), while stirring constantly, into 400 g. of methanol (99.5 per cent). Preserve in a glass-stoppered bottle.

(*c*) *Potassium Hydroxide in Methanol.*—Dissolve 33 g. of potassium hydroxide, preferably in the pellet form, in 1 liter of methanol (99.5 per cent) to form an approximately half normal solution.[14] Potassium acid phthalate is preferable for standardizing the solution; thus 2.553 g. of $C_6H_4COOKCOOH$ will be neutralized by 25.00 ml. of 0.5000 *M* KOH.

(*d*) *Thymol Blue Solution.*—Dissolve 0.10 g. of thymol blue in 100 ml. of methanol.

Procedure

25. Weigh 4 to 5 g. of the tall oil sample in a tared 250-ml. conical flask on a scale having a sensitivity of 0.01 g. Add 150 ml. of methanol, and twirl the flask to dissolve the oil. Add 5 ml. of the methyl sulfuric acid solution and boil the entire mixture for 2 min. under a reflux condenser. When the contents have cooled, add 1 ml. of the thymol blue solution and titrate with the methanol solution of potassium hydroxide. Continue this titration until the thymol blue turns from red to yellow (about pH of 3); note the reading and then refill the burette if necessary. Continue the titration with the potassium hydroxide until the thymol blue turns from yellow to blue (about pH of 9). The milliliters of 0.5 *M* KOH consumed between the two end points are equivalent to the rosin acids present.

Calculation

26. Calculate the rosin acids number (Note) from the following formula:

$$\text{Rosin acids number} = \frac{A \times M \times 56.1}{W}$$

where:

A = milliliters of KOH consumed between the two ends points,

M = molar concentration or normality of KOH solution, and

W = weight of sample in grams, dry basis.

NOTE.—The rosin acids number is convenient for calculations and corresponds to an acid number in that it expresses the milligrams of KOH consumed by the neutralization of the rosin acids in 1 g. of the sample.

Alternate McNicoll Method[15]

Apparatus

27. The apparatus shall consist of the following:

(*a*) *Esterification Flask.*—A 250-ml. wide neck flask, standard 34/45 taper, of the Erlenmeyer type.

(*b*) *Reflux Condenser.*—Any suitable water-cooled, glass reflux condenser.

(*c*) *Titration Stage.*—A specially constructed case, with an Eastman color filter, consisting of a Wratten "C" No. 52, mounted in B glass, 3 in. square, protected on both sides by a Corning $3\frac{1}{8}$ in. square polished glass, 2 mm. in thickness, medium shade, AKLO 397. This combination of glasses is seated in a cut-out section in the center of the top of the case. The interior shall be illuminated by electric lamps, in each end of the case, concealed from the view of the analyst. The light shall be reflected from a white opaque glass plate, placed in the bottom of the case. The dimensions of the case shall be: top and bottom 14 by 6 in.; sides, 14 by 5 in.; ends, 6 by 5 in.

Reagents

28. (*a*) *Potassium Hydroxide (0.2 N).*—Standardize a 0.2 *N* solution of KOH in 99.5 per cent methyl alcohol. Due to the volatility of alcohol, this solution should be standardized frequently.

(*b*) *Thymol Blue Solution.*—Dissolve 0.1 g. of thymol blue in 100 ml. of methanol.

[15] Modified from the Standard Methods of Sampling and Chemical Analysis of Soaps and Soap Products (A.S.T.M. Designation: D 460), see 1944 Book of A.S.T.M. Standards, Part III.

(*c*) *Naphthalene-β-Sulfonic Solution.*—Dissolve 20 g. of Eastman grade or the equivalent in 1 liter of c.p. absolute (or 99.5 per cent) methyl alcohol.

Procedure

29. (*a*) *Esterification.*—Weigh out about 2 ± 0.1 g. of tall oil into the esterification flask. Add 25 ml. of naphthalene-β-sulfonic acid solution. Add a few glass beads to insure smooth boiling, attach the reflux condenser, and boil gently for 30 min. Run a blank using 25 ml. of the reagent alone. At the end of the boiling period remove the flask and cool.

(*b*) *Titration.*—Add 1 ml. of thymol blue indicator and titrate with 0.2 *N* alcoholic KOH. Titrate with the base of the flask resting on the color filter plate, and observe vertically. As the test solution darkens on approaching the end point (pH of 9.6), add the standard solution one drop at a time, observing whether or not a dark blue cloud forms at the surface where the drop comes in contact with the test solution. The end point is that at which the addition of one more drop of standard alkali solution causes no visible cloud or darkening at the instant and point where it falls into the solution, and produces no streaks as it disperses.

Calculation

30. The rosin acids number of tall oil when determined by the modified McNicoll method shall be calculated as follows:

$$R_1 = \frac{(T - B) \times M \times 56.1}{W} - 1$$

where:

R_1 = rosin acids number, corrected (Note),
T = milliliters of KOH required to titrate sample,
B = milliliters of KOH required to titrate blank,
M = molar concentration or normality of KOH solution, and
W = weight of sample, in grams, dry basis.

NOTE.—Cooperative studies on rosin in soaps have indicated that the McNicoll method gives results approximately 1 per cent higher than those obtained by other methods for rosin content of soap. The committee therefore recommends tentatively that in reporting the rosin acid number of tall oil the results by the McNicoll method be corrected by subtracting 1 from the recorded or calculated figure.

ROSIN ACIDS

Procedure

31. The percentage of rosin acids in the tall oil shall be calculated as abietic acid as follows:

$$\text{Rosin acids, per cent} = \text{Rosin acids number} \times \frac{302}{561}$$

where:

302 = molecular weight of abietic acid.

QUALITATIVE TEST FOR ROSIN

Procedure

32. In any case where the rosin acid content is found to be less than 5 per cent, the actual presence of rosin should be checked qualitatively by the Liebermann-Storch test, as follows: Transfer about 2 ml. of the tall oil to a test tube, add 5 ml. of reagent grade acetic anhydride, and dissolve by warming on a steam bath or by other gentle heating. Cool and pour about one half the solution into a small white porcelain evaporating dish. Allow one drop of H_2SO_4 (sp. gr. 1.53) to run down the side of the dish. To prepare this special sulfuric acid, dilute 34.7 ml. of concentrated acid (sp. gr. 1.84) with 35.7 ml. of distilled water. If rosin acids are present a bright but fugitive violet coloration appears where

the liquids come into contact, which changes rapidly to a brownish tinge. It is advisable to make a check test with a sample of fatty acid to which a small quantity of rosin or rosin acid has been added.

UNSAPONIFIABLE MATTER (STEROLS, HIGHER ALCOHOLS), ETC.[16]

Reagents

33. (*a*) *2.0 M Potassium Hydroxide in Methanol* (*99.5 per cent*).

(*b*) *Diethyl Ether*.

Procedure

34. Weigh 5.00 g. of the tall oil in a small Erlenmeyer flask, add 12 to 15 ml. of the potassium hydroxide solution, and reflux for 1.5 hr. Transfer the contents of the flask to a separatory funnel with the aid of about 50 ml. of water, and extract with ether, using successive portions of 40, 30, 30, and 30 ml. Collect the ether extracts in a separatory funnel containing 20 ml. of water. Run off the water without shaking the funnel. Then wash the ether solution by shaking it vigorously with 2, 5, and 30 ml. of water. Transfer the solution to a small tared beaker, evaporate, and dry at 100 to 105 C. to constant weight. Calculate the percentage of unsaponifiable matter (sterols, higher alcohols, etc.), using the weight of sample corrected to the dry basis.

FATTY ACIDS

Procedure

35. Calculate the fatty acids content of the tall oil as follows:

$$\text{Fatty acids, per cent} = 100 - (R + U)$$

where:

R = percentage of rosin acids, and
U = percentage of unsaponifiable matter.

[16] This method is taken from Wilkie's "Estimation of Unsaponifiable Matter in Oils, Fats, and Waxes," *Analyst*, Vol. 42, p. 200 (1917). Also described in Allen's *Commercial Organic Analysis*, Part II, Vol. 90 (5th edition).

Tentative Definitions of
TERMS RELATING TO TALL OIL[1]

A.S.T.M. Designation: D 804 – 44 T

ISSUED, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Many definitions have been proposed from time to time for tall oil and associated materials. Great care is desirable in devising tall oil definitions that might become established. Any categories for tall oil should be acceptable to all tall oil producers and consumers.

Aside from the academic need for suitable definitions, the problem has commercial importance. A confused technology impairs sales and invites unfair trade practices. It has also resulted in unfair freight rates.

It should not be difficult to devise clear definitions for the principal tall oil materials. There is some question as to the value of a detailed classification of tall oil products not yet in commerce.

Definitions should avoid so far as possible any specifications or "grade labeling." Thus tall oil is now being offered as crude tall oil, refined tall oil, and semirefined tall oil. Rather than attempt to define these materials, it seems desirable to define only tall oil and provide separately a series of specifications for the various qualities.

The following definitions should prove acceptable from this point of view.

Rosin Acids.—The organic acids in tall oil that respond to the Liebermann-Storch test for rosin and that remain unesterified after the application of the Wolff method or similar esterification procedures. The molecular weight of these acids shall be considered to be 302.

Skimmings.—The curd, not acidified or otherwise processed, skimmed from the black liquor of the alkaline paper pulp industry.

Tall Oil.—The natural mixture of rosin acids related to abietic acid, and of fatty acids related to oleic acid, together with nonacidic bodies, which is the product of the acidification of the skimmings from the black liquor of the alkaline paper pulp industry.

Tall Oil Abietic Acid.—The crystalline or solid product separated by crystallization from tall oil consisting principally of rosin acids.

Tall Oil Pitch.—The undistilled residue from the distillation of tall oil.

Tall Oil Soap.—The product formed by the saponification or neutralization of tall oil with organic or inorganic bases.

EXPLANATORY NOTE

NOTE.—The following designations for tall oil shall be considered obsolete:

Crude resinous liquid	Sulfate rosin
Finn oil	Swedish pine oil
Lignin liquor	Swedish resin
Liquid resin	Swedish rosin
Liquid rosin	Swedish resin oil
Pine wood pitch	Swedish rosin oil
Resin oil	Sylvic oil
Sulfate pitch	Talloel
Sulfate resin	Tallol

[1] Under the standardization procedure of the Society, these definitions are under the jurisdiction of the A.S.T.M. Committee D-17 on Naval Stores.

[2] Accepted by the Society at annual meeting, June, 1944.

Tentative Specifications for

ISOPROPYL ALCOHOL[1]

A.S.T.M. Designation: D 770 – 44 T

ISSUED, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These specifications cover isopropyl alcohol (99 per cent grade).

Properties

2. Isopropyl alcohol (99 per cent grade) shall conform to the following requirements:

Specific gravity, 20/20 C	0.785 to 0.790
Color	water white
Distillation range	not greater than 1.5 C. and this range shall include 82.3 C.
Nonvolatile matter	not more than 0.005 g. per 100 ml.
Odor	mild, nonresidual
Water	miscible without turbidity with 19 volumes of 60° A.P.I. gasoline at 20 C.
Acidity	not more than 0.02 mg. of KOH per gram of sample.
Hydrocarbons	miscible without turbidity with 10 volumes of distilled water at 20 C.

Methods of Testing

3. The material shall be sampled and the properties enumerated in these specifications determined in accordance with the Standard Methods of Sampling and Testing Lacquer Solvents and Diluents (A.S.T.M. Designation: D 268).[3]

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Accepted by the Society at annual meeting, June, 1944.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Tentative Specifications for

METHYL ETHYL KETONE[1]

A.S.T.M. Designation: D 740 – 43 T

ISSUED, 1943.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These specifications cover methyl ethyl ketone.

Properties

2. Methyl ethyl ketone shall conform to the following requirements:

Purity	as agreed upon by the seller and the purchaser
Specific gravity, 20/20 C.	0.805 to 0.807
Color	water white
Distillation range:	
Below 78 C.	none
Above 81 C.	none
Nonvolatile matter	not more than 0.005 g. per 100 ml.
Odor	characteristic, nonresidual
Water	miscible without turbidity with 19 volumes of 60° A.P.I. gasoline at 20 C.
Acidity (free acid as acetic acid)	not more than 0.003 per cent by weight, equivalent to 0.028 mg. of KOH per gram of sample.

Methods of Testing

3. The material shall be sampled and the properties enumerated in these specifications determined in accordance with the Standard Methods of Sampling and Testing Lacquer Solvents and Diluents (A.S.T.M. Designation: D 268).[3]

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Accepted by Committee E-10 on Standards, August 30, 1943.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Tentative Method of Test for

WATER IN LACQUER SOLVENTS AND DILUENTS[1]

A.S.T.M. Designation: D 268 - 44 T

ISSUED, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. (*a*) This method of test covers the procedure for the determination of water present in any proportion in most organic liquids, such as lacquer solvents and diluents.[3]

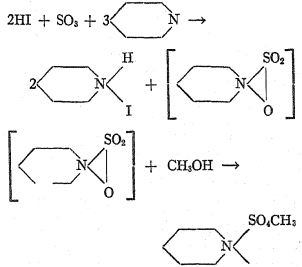
(*b*) Carbonyl compounds and primary, secondary, and certain tertiary amines react slowly with the reagents causing a fading end point and consequently erratic results. This procedure has been modified to include such compounds and to eliminate these undesirable side reactions.

Principle of Test

2. (*a*) In the presence of water, iodine and sulfur dioxide react as follows:

$$I_2 + SO_2 + H_2O \leftrightarrows 2HI + SO_3$$

(*b*) The iodine and sulfur dioxide are dissolved in anhydrous methanol to which is added sufficient pyridine to neutralize the HI and SO_3 formed by the following reaction:



(*c*) When a solution containing water is titrated with the reagent the color fades instantaneously until all the water is consumed. The persistence of the brown iodine color indicates the end point of the reaction.

Reagents

3. *Iodine - Sulfur Dioxide.*—Prepare the iodine - sulfur dioxide as follows by

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] This method is in effect a tentative revision of and, when adopted, is intended to be added to the Standard Methods of Sampling and Testing Lacquer Solvents and Diluents (A.S.T.M. Designation: D 268 - 44), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

[3] This procedure has been adapted from the method of Karl Fischer published in *Angewandte Chemie*, Vol. 48, pp. 394–396 (1935); *Chemical Abstracts*, Vol. 29, No. 6532 (1935), and from further observations by Smith, Bryant, and Mitchell published in the *Journal*, Am Chemical Soc., Vol. 61, p. 2407 (1939).

dissolving the listed materials in the order shown:

Methanol, substantially dry	5 l.
Iodine	254 g.
Pyridine, dry, freshly distilled	790 g.
Sulfur dioxide, dried by passing through H_2SO_4 (sp. gr. 1.84)	192 g.

NOTE.—The reagent rapidly loses strength when exposed to the air and it is best preserved and used in an automatic burette.[4] All vents open to the air must have protective calcium chloride tubes. Much of the accuracy of the method is lost if an ordinary burette is used without special precautions.

Standardization of Reagent

4. (*a*) Place 10 to 15 ml. of substantially dry methanol in a 125-ml. glass-stoppered Erlenmeyer flask which has been carefully dried. Titrate the alcohol with the reagent until the brown color of the iodine persists. Shake the contents of the flask vigorously near the end of the titration and continue titrating until the color change is permanent. Keep the flask stoppered as much as possible as there is sufficient moisture in the air to fade the end point (Note).

NOTE.—During the titration the solution turns yellow after the addition of the first few milliliters of the reagent. This color change must not be confused with the true end point. Although the yellow color deepens towards the end of the titration, the transition from yellow to brown is quite sharp and easily reproducible.

(*b*) Weigh 0.05 to 0.08 g. of distilled water (about 2 to 4 drops) into the flask from a Grethen or similar weighing pipette. Again titrate to the same brown end point. Make several check determinations.

(*c*) Standardize the reagent daily to obtain the best results. Calculate the factor for the reagent in grams of water consumed by 100 ml. of reagent as follows:

$$A = \frac{100B}{C}$$

where:

A = water consumed in grams by 100 ml. of reagent,
B = water in grams used in standardization, and
C = milliliters of reagent required for titration.

Sample

5. (*a*) Use a portion of the sample such that the amount of water present corresponds to 40 to 45 ml. of the reagent. The amount of sample to be used can best be estimated by a previous knowledge of the approximate water content or by a preliminary trial.

NOTE.—If absolutely dry materials were available for the preparation of the reagent the theoretical factor would be about 0.36. Actually, the factor is usually about 0.18 due to small quantities of water in the reagents. The following table gives a few typical examples of suitable samples to use for reagents having a factor of 0.16 and 0.20. Such samples will give a titration of 40 ml.

Water, per cent	Size of Sample, g. 0.16 Factor	0.20 Factor
0.2	32.0	40.0
1	6.4	8.0
10	0.64	0.8
50	0.13	0.16
80	0.08	0.1
100	0.06	0.08

These amounts may be increased as much as 20 per cent, giving a titration of 48 ml. or decreased to a sample size convenient for pipetting or weighing.

(*b*) Samples having a volume of more than 1 ml. may be measured volumetrically with an accurately calibrated pipette. Weigh all smaller samples.

Procedure

6. (*a*) Place 10 to 15 ml. of methanol in a 125-ml. glass-stoppered Erlenmeyer flask and titrate to a brown end point, observing the precautions given in Section 4. Add the sample and again titrate

[4] A type similar to model 1955 manufactured by the Scientific Glass and Apparatus Co., Bloomfield, N.J., may be used.

with the reagent to the same brown end point.

(*b*) When determining water in the presence of carbonyl compounds replace the methanol with 25 ml. of substantially dry, freshly distilled pyridine.

(*c*) In the presence of amines, use 10 to 15 ml. of reagent grade glacial acetic acid in place of the methanol. Use 50 ml. of the acid if more than 10 ml. of sample is to be used.

Calculation

7. Calculate the percentage by weight of water present as follows:

$$W = \frac{A \times C}{D}$$

where:

W = percentage by weight of water present,
A = water in grams consumed by 100 ml. of reagent,
C = milliliters of reagent required for titration, and
D = grams of sample used.

Tentative Method of Test for

DRY TO NO-PICK-UP TIME OF TRAFFIC PAINT[1]

A.S.T.M. Designation: D 711 - 43 T

ISSUED, 1943.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. This method describes a laboratory test to determine the length of drying time after application for no-pick-up of traffic or pavement-marking paint by the tire of an automobile.

Apparatus

2. The apparatus as shown in Fig. 1 shall consist of a small, rubber-tired wheel with roller bearings, mounted on an axle so that it rotates freely. The axle shall be fitted with knob handles, and shall be equally weighted on each side. The total weight of the apparatus shall be 35 lb. The detailed dimensional requirements shall conform to those shown in Fig. 1.

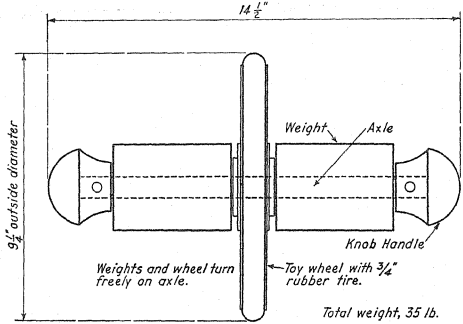


FIG. 1.—Weighted Wheel for Dry to No-Pick-Up Time Test of Traffic Paint.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Accepted by the Society at annual meeting, June, 1943.

Procedure

3. (*a*) A test strip at least 3 in. in width of the paint to be tested shall be applied by a mechanical spreader, brush, or other suitable means to a clean plate glass panel at a wet film thickness of 0.004 in. The time of application shall be recorded. The panel shall be allowed to dry in a horizontal position under the laboratory conditions specified (preferably 25 C. and 50 to 60 per cent relative humidity).

(*b*) At regular intervals, the weighted wheel shall be rolled over the paint film. The end point for dry to no-pick-up time shall be noted when no paint adheres to the rubber tire of the test wheel when it is rolled over the paint film. As the end point is approached the weighted wheel shall be rolled over the paint film every 30 sec. The time elapsed between application of the paint and the end point shall be recorded as the drying time for no pick-up of the traffic paint.

Tentative Method of Test for

LIGHT SENSITIVITY OF TRAFFIC PAINT[1]

A.S.T.M. Designation: D 712 - 43 T

ISSUED, 1943.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. This method of test is intended for determining the color change produced by sunlight on paint material intended for use as traffic or pavement-marking paint.

Preparation of Test Panels

2. (*a*) *Preferred.*—Two test films of the traffic paint to be tested and two films of a mutually agreed upon standard paint shall be prepared. The films shall be prepared on clean, smooth plate glass panels. The paint films shall be at least 1½ by 3 in. and of uniform and sufficient thickness to provide complete hiding. It is recommended that the films be applied by a Bradley or other suitable mechanical spreader. Following application, the films shall be dried for a period of 24 hr. under normal room conditions (approximately 25 C. and 50 per cent relative humidity).

(*b*) *Alternate.*—Two test films of the traffic paint to be tested shall be applied to panels with a flat opaque diffusing surface. The films shall be at least 1½ by 3 in. and of uniform and sufficient thickness to provide complete hiding. The films shall be applied and dried as described in Paragraph (*a*).

Procedure

3. (*a*) *Preferred.*—One test panel of the paint to be tested and of the mutually agreed upon standard paint prepared as described in Section 2 (*a*) shall be exposed without water spray for a period of 4 hr. at equal distances (15 or 18 in.) from an enclosed type carbon arc light conforming to the Tentative Recommended Practice for Characteristics of Standard Carbon Arc Accelerated Weathering Unit (A.S.T.M. Designation: E 42) of the American Society for Testing Materials.[3] Immediately after completion of the exposure, each exposed film shall be compared for change in brightness or discoloration with the corresponding duplicate unexposed film. The test paint shall not show greater loss in brightness nor greater discoloration than the standard.

(*b*) *Alternate.*—One of the test panels prepared as described in Section 2 (*b*)

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Accepted by the Society at annual meeting, June, 1943.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

shall be exposed without water spray for a period of 4 hr. at a distance of 15 or 18 in. from an enclosed type carbon arc light conforming to the Tentative Recommended Practice E 42. The brightness of the test paint film before and after the exposure shall be measured by a suitable instrument, such as the Hunter multipurpose reflectometer.[4]

[4] This instrument is described in a paper by Richard S. Hunter, "A Multipurpose Photoelectric Reflectometer," *Journal of Research*, Nat. Bureau Standards, Vol. 25, No. 5, November, 1940, p. 581. (*Research Paper, RP1345.*)

Tentative Method of

CONDUCTING ROAD SERVICE TESTS ON TRAFFIC PAINT[1]

A.S.T.M. Designation: D 713 – 43 T

ISSUED, 1943.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. This method of test is intended for determining the relative values of service of traffic or pavement-marking paints under actual road conditions. Samples of the paints being tested are compared under prescribed conditions and periodic observations are made as to the relative performance characteristics as a basis of comparison.

Type of Location of Road Surface for Tests

2. (*a*) The test area or areas shall be selected as being representative of the road surface upon which the paint actually will be used. These surfaces may include portland-cement concrete, sheet asphalt, bituminous concrete, rock asphalt, and bituminous surface treatment.

(*b*) The location of test areas shall be such that the traffic shall flow uninterruptedly over the entire length of test area. Proximity of the test area to curves, change of grade, intersections, stop signals, or other traffic diversion or control devices shall be avoided. Test stripes shall be located in areas with normal pavement drainage and where they will not be shaded by high banks, trees, or buildings.

(*c*) In order to accelerate the test, the traffic in vehicles per unit time over the test area shall be at least equal to average traffic conditions. If desired, average traffic counts may be taken during the tests.

Conditions at Time of Application

3. The air temperature at the time of application of the samples of paint shall be normal summer temperature, preferably not less than 60 nor more than 90 F. To insure normal pavement testing conditions, the surface shall be free of moisture when the paints are applied; application between 10 a.m. and 3 p.m. is recommended.

Consistency of Paint Samples

4. (*a*) When road tests are to be made with samples for which no control paint has been specified or mutually agreed upon, these samples shall be applied as furnished unless the supplier

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Accepted by the Society at annual meeting, June, 1943.

provides specific instructions for adjusting the consistency, in which case the supplier shall also furnish the thinners required by the instructions.

(*b*) If comparison is to be made with a control paint, the consistency of the test sample shall not, unless otherwise mutually agreed upon, vary more than plus or minus 5 per cent from the consistency of the control paint as measured by a mutually agreeable method of test, preferably the Standard Method of Test for Consistency of Exterior House Paints and Enamel-Type Paints (A.S.T.M. Designation: D 562) of the American Society for Testing Materials.[3]

Method and Rate of Application

5. (*a*) The paints shall be applied by brush, spray, or mechanical applicator, the specific method of application to be at the option of the testing agency. Particular care shall be taken in application by brush on bituminous surfaces so as not to cause premature bleeding of the bitumen through the paint film by excessive or vigorous use of the brush. It is recommended that the width of the test stripe be the same as applied in actual service except that it shall not be less than 4 in. in width. The stripes shall be applied either perpendicularly or diagonally to traffic (Note 1). The stripes shall be applied conveniently close together to facilitate observations.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(*b*) The rate of application shall be at the option of the testing agency. It is recommended, however, that on portland-cement concrete the spreading rate shall be not less than 125 nor more than 250 sq. ft. per gal. (Note 2). The spreading rate of the samples being tested shall be the same, except that a tolerance of plus or minus 5 per cent variation in spreading rate will be permissible.

NOTE 1.—If tests are conducted on a broomed concrete pavement, the stripes should be applied diagonally to traffic, preferably 45 deg. to the direction of the brooming.

NOTE 2.—These spreading rates are equivalent to a range of 7 to 14 gal. per mile for a stripe 4-in. in width and 11½ to 21 gal per mile for a stripe 6-in. in width.

Observations

6. (*a*) Inspection of the test sections shall be made at regular intervals. The following schedule of inspections is recommended:

First inspection	immediately prior to subjecting the test stripe to traffic
Second inspection	24 hr. after application
Third inspection	2 weeks after application
Fourth inspection	4 weeks after application
Fifth inspection	8 weeks after application
Subsequent inspections	every 4 weeks thereafter

(*b*) Observations shall be made as to the relative performance characteristics, such as general appearance, day and night visibility, bleeding, chipping, and abrasion. It is recommended that photographs be made as a supplementary record of the relative performance characteristics.

Tentative Method of Test for

SPECULAR GLOSS OF PAINT FINISHES[1]

A.S.T.M. Designation: D 523 - 44 T

ISSUED, 1939; REVISED, 1941, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. This method of test describes three procedures for measuring specular gloss of paint finishes as follows:

Procedure A may be used for the classification of any surface regardless of the presence of some texture and may also be used for measurement of eggshell and flat finishes.

Procedure B is applicable only to surfaces which are free from macroscopic structure or texture and is intended for measurement of specular gloss over a wider gloss range than procedure A.

Procedure C may be used for the routine comparison of the gloss of surfaces of low and medium gloss where all surfaces in any group have approximately the same diffuse reflectance.

NOTE.—Properties other than specular gloss that contribute to the appearance of a finish are defined in Appendix I and discussed in Appendix II.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Latest revision accepted by the Society at annual meeting, June, 1944.

Definition

2. *Specular Gloss.*—Specular gloss is defined as one thousand times the ratio of the brightness of the sample when illuminated unidirectionally[3] and viewed in the direction of specular reflection less the brightness of the sample when illuminated in the same direction and viewed normally,[4] to the brightness of the ideal, completely reflecting, perfect mirror, similarly illuminated and viewed in the direction of specular reflection.

Apparatus

3. (*a*) The apparatus shall consist of a light source, means for locating the surfaces of test objects, and a receptor. These three parts shall be combined in a rugged instrument with properly collimated source and receptor beams. The proper relative positions of the dif-

[3] When the source subtends at the sample a solid angle so small that a further decrease will not alter appreciably either the angular distribution of the reflected flux or its ratio to the incident flux, the illumination is called unidirectional.

[4] Interchange of source and receptor is permissible. It should be noted that procedure C does not measure the quantity here defined because it does not provide for subtracting the brightness of the sample when viewed normally from the brightness when viewed in the direction of specular reflection. As noted in Section 1, however, procedure C has been designed for the rapid gloss comparison of surfaces of approximately the same diffuse reflectance.

ferent parts of such an instrument are shown in Fig. 1.[5]

(*b*) *Angles of Illumination and View.*—The angle of incidence shall be that prescribed in Section 4 (*a*) or (*b*). The direction of view shall be that of mirror reflection. With a front-surfaced mirror in a testing position, the reflected beam shall be centered on the receptor. Provision shall also be made for viewing or illuminating the sample normally to its surface, except in instruments designed only for measurements according to procedure C.

(*c*) *Spectral Characteristics of Source and Receptor.*—The source shall approximate standard illuminant *C* (representative of average daylight) recommended by the International Commission on Illumination (I.C.I.), and the receptor shall have a spectral sensitivity approximating the luminosity factors of the standard I.C.I. observer, or equivalent source-receptor combinations may be used.

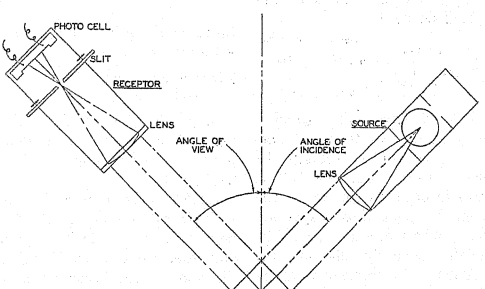


FIG. 1.—Drawing of Glossmeter Showing Arrangement of Parts.

Method of Measurement

4. (*a*) *Procedure A.*—To classify surfaces according to specular gloss, measurements[6] shall be made at 60 deg. with the following rectangular source and receptor spreads:

	Source	Receptor
Aperture spread in the plane of the centers of the incident and reflected beams	0.6 deg.	4.4 deg.
Aperture spread perpendicular to the plane of the centers of the incident and reflected beams	3.7 deg.	11.7 deg.

Interchange of source and receptor spreads or the use of different spreads is permissible, provided specular gloss values obtained do not differ from those measured according to the prescribed conditions by more than plus or minus 2 for specular gloss values above 20, or by more than plus or minus 10 per cent for values equal to or below 20. When readings in the direction of specular reflection are less than 20, each reading shall be diminished by an amount which will compensate for the light diffusely reflected from the sample. This may be accomplished by making an additional measurement with the source normal to the sample and the receptor in the same position as before.[7]

[5] Constructions differing from that specified may be employed if such constructions are proved to give identical results.

[6] Richard S. Hunter and Deane B. Judd, "Development of a Method of Classifying Paints According to Gloss," ASTM BULLETIN, No. 97, March, 1939, p. 11.

[7] In lieu of determining the compensation in the above manner, it may be determined by multiplying the apparent reflectance, $A_{45°,\pm\epsilon}$ by 2.5 (for most paint films either $A_{45°,0°}$ or $A_{diffuse,0°}$ may be substituted for $A_{45°,0°}$), when instruments employing the source and receptor spreads specified above are used.

When measured under these conditions, surfaces shall be classified as follows:

Surfaces Having Gloss Values	Classed As	Usually Referred to As
Above 70	A	High or full gloss
70 to 30, excl.	B	Semigloss or medium gloss
30 to 6, excl.	C	Eggshell
6 to 2, excl.	D	Flat to eggshell (intermediate)
2 and under	E	Flat

(*b*) *Procedure B.*—For the measurement of specular gloss the following conditions shall be used:

Gloss Range	Angle of Incidence and View, deg.
High and medium or semigloss	45
Eggshell	60
Flat	70

For measurement of the specular gloss, the following apertures for the source and receptor shall be used:

	Source	Receptor
Aperture in the plane of the collimator and receptor axes	0.5 deg.	1.5 deg.
Aperture in the plane perpendicular to the collimator and receptor axes	3.5 deg.	4.5 deg.

For samples classified as flat an additional measurement with the source normal to the sample shall be made with the same viewing angle and apertures.[4] This is necessary in order to correct for the diffusely reflected light whose magnitude in the case of such samples may be comparable to the specularly reflected light. This additional measurement must be deducted from the measurement made with the source located at the position specified above for the determination of the mirror reflection, in order to determine the specular gloss.[8]

(*c*) *Procedure C.*—For measurements of specular gloss according to procedure C, the 60-deg. illuminating and −60-deg. viewing conditions specified for procedure A shall be used. Values of specular gloss according to procedure C are therefore always higher than values for the same specimens according to procedure A by the amounts of the diffuse corrections required by procedure A.

[8] This procedure should also be carried out for intermediate, eggshell, medium, and high-gloss samples, if necessary.

Specular Gloss Standards

5. (*a*) The fundamental specular gloss standard shall be the ideal, completely reflecting, perfect mirror which is assigned a value of 1000.

(*b*) Specular-gloss working standards may be highly polished, plane, black-glass surfaces for which the reflectance may be calculated as a function of angle of incidence and the refractive index by using Fresnel's equation. Polished black glass with a refractive index of 1.52 has the following values for the several angles of incidence shown:

Angle of Incidence, deg.	Specular Gloss
45	53
60	92.5
70	170

Surfaces of liquids may also be used[9] as working standards.

Preparation and Measurement of Samples

6. (*a*) Samples of the finish to be tested shall be prepared on smooth, plane panels with or without a ground as may be desired.

(*b*) For measurement in accordance with procedures A or C, the finish shall be as free as possible from brush marks and other surface nonuniformities. The direction of the brush marks or other similar texture effects shall be parallel to the plane of measurement.

For measurement in accordance with procedure B, the finish shall be free from brush marks, orange peel, spray wave, and other surface irregularities.

(*c*) In making the measurements, check-settings on a reference standard which has been calibrated for use under like conditions shall be interspersed at time intervals sufficiently brief to insure accuracy of the results to within 5 per cent.[10]

[9] D. G. Moore and R. S. Hunter, "Use of Liquid Surfaces as Standards of Specular Gloss," *J. Am. Ceramic Soc.*, Vol. 24, p. 167 (1941).

[10] For determining the specular gloss of specimens similar in reflecting characteristics to an available standard, larger apertures yield equally accurate results which are often more precise.

(*d*) At least three portions of the surface of the test specimen shall be measured to give an indication of the degree of surface uniformity and provide an average which is typical of the surface. If any portion of the test surface differs from the average in specular gloss by more than 5 per cent the panel shall, unless otherwise agreed, be discarded.

Report

7. (*a*) Unless otherwise specified, the value of specular gloss shall be reported in terms of a perfectly reflecting mirror which has been assigned a value of 1000.

(*b*) The angle of incidence shall be reported.

(*c*) The method of preparation and the film thickness shall be stated whenever possible.

APPENDIX I

DEFINITIONS OF FIVE TYPES OF GLOSS NOT INVOLVED IN PRESENT METHOD OF TEST

A1. (*a*) *Gloss.*—Gloss is the property of a surface by which it reflects light specularly.

(*b*) *Glossiness.*—Glossiness of a surface is the appearance ascribable to its gloss.

(*c*) *Apparent Reflectance.*—Apparent reflectance of a sample is the reflectance which a perfectly diffusing surface would require in order to yield the same brightness as the sample under the same conditions of illumination and viewing.

(*d*) *Sheen.*—Sheen is specular gloss at near grazing angles of incidence and reflection.

(*e*) *Contrast Gloss.*—Contrast gloss is expressed as a fraction whose denominator is the apparent reflectance of the sample when illuminated in a direction other than perpendicular and viewed in the direction of specular reflection, and whose numerator is the same apparent reflectance diminished by the apparent reflectance of the sample identically illuminated but viewed perpendicularly.

(*f*) *Distinctness-of-Image Gloss.*—Distinctness-of-image gloss is indicated by the faithfulness with which the surface produces mirror images of objects.

(*g*) *Absence-of-Bloom Gloss.*—Absence-of-bloom gloss is indicated by freedom from haziness bordering a high light.

(*h*) *Surface-Uniformity Gloss.*—Surface-uniformity gloss is revealed by the degree of freedom of relatively high-gloss surfaces from visible local variations.

APPENDIX II

DISCUSSION OF DEFINITIONS

A2. (*a*) *Apparent Reflectance.*—In the measurement of gloss one deals mainly with unidirectional illumination; for such illumination apparent reflectance is a function of the altitude and azimuth of the illuminating beam, and the altitude and azimuth of the direction of view. The gloss of a surface is completely specified by these unidirectional apparent reflectances and is the degree to which apparent reflectance varies with angle. Of principal importance are the specular and near-specular directions of reflection.

In actual cases six determinations corresponding to the six types of gloss defined above have been found sufficient. Five of these may be expressed as functions of apparent reflectance in the plane of the illuminating beam and normal to the surface using the symbol $A_{\theta,\theta'}$, for apparent reflectance where the first subscript indicates the direction of the illuminating beam measured from the normal, and the second subscript similarly indicates the direction of view.

(*b*) *Specular Gloss, Sheen.*—The reference standard used in calculating specular gloss or sheen is the perfectly reflecting mirror. The working standard is customarily a piece of polished black glass whose specular reflectance is calculated by the Fresnel law from its index of refraction. If angle of illumination and the sizes of illuminating and viewing apertures are

stated, specular gloss (G_s) and sheen (G_{sh}) are given by the formula:

$$G_s \text{ or } G_{sh} = 1000 \left(\frac{B_{\theta,-\theta} - B_{\theta,0}\,(\text{sample})}{B_{\theta,-\theta}\,(\text{standard})} \right)$$

Where:

$B_{\theta,-\theta}$ = brightness for equal angles of viewing and illumination, and

$B_{\theta,0}$ = brightness for the same angle of illumination and normal viewing (conditions of viewing and illumination may be reversed).

(*c*) *Contrast Gloss.*—If angle of illumination and sizes of illuminating and viewing apertures are stated, contrast gloss, G_c, is given by the formula:

$$G_c = \frac{(A_{\theta,-\theta} - A_{\theta,0})}{A_{\theta,-\theta}}$$

(*d*) *Distinctness-of-Image Gloss.*—Distinctness-of-image gloss depends on the rate of change with angle of incidence of apparent reflectance for a direction of view deviating from the direction of specular reflection by a small angle (perhaps from 1 min. to 1 deg.). A possible quantitative definition of this type of gloss G_i is:

$$G_i = \frac{\dfrac{dA_{i,-\theta}}{di}}{k + \dfrac{dA_{i,-\theta}}{di}}$$

where:

$\theta = i + \delta i$

δi = a small angle of the order of 1 min. to 1 deg., and

k = an arbitrary constant which should be adjusted once for all to give a convenient scale.

Distinctness-of-image gloss may also be expressed as a function of the least angular separation, $\delta\theta$, of two linear elements in the illuminant which are resolved in the image. This might give:

$$G_i = \frac{K}{K + \delta\theta}$$

(*e*) *Absence-of-Bloom Gloss.*—Bloom may be exhibited by surfaces whose distinctness-of-image gloss is high and may be quantitatively described by the difference between the apparent reflectance of the specimen a few degrees $\delta\phi$ (perhaps 5 deg.) from the direction of mirror reflection and the apparent reflectance many degrees $\Delta\phi$ (perhaps 45 deg.) from it. Absence-of-bloom gloss (G_b) might be indicated by:

$$G_b = \frac{k}{[k + (A_{\theta,-(\theta-\delta\phi)} - A_{\theta,-(\theta-\Delta\phi)})]}$$

Bloom is haziness near a high light. It may be caused by a smear of foreign substance (such as grease) on a plane surface, by small discontinuities in the surface itself, or by light-scattering elements beneath the surface. Bloom from the first two sources is usually an undesirable quality because it may be the indication of a cleaning process not quite successful, or of the incipient breakdown of a glossy paint film. Bloom from the third source, is, however, frequently a desired quality, and light-scattering materials are sometimes introduced for the purpose of producing it. Such a paint is sometimes said to have a "satiny" finish.

(*f*) *Surface-Uniformity Gloss.*—The dependence of glossiness upon the presence of surface nonuniformities is definite for samples of high gloss, less definite for surfaces of eggshell finish, and obscure or absent for surfaces of nearly flat finish. Surface nonuniformities probably influence judgments of distinctness-of-image gloss more than judgments of other types. Surface nonuniformities include both the regular or semiregular nonuniformities sometimes called texture (brush marks, orange peel, marks exhibited by etched, pitted, scratched, or wavy surfaces and so on) and the irregular nonuniformities (blotches, isolated scratches, pits, waves, cracks, lines or spots, and so on). Thus far, surface nonuniformity has been treated by visual judgments and by photographs which serve to record the amount and kind of nonuniformity.

(*g*) *Characteristic Appearance of Each Type of Gloss, and Range in which Each Is Most Prevalent:*

Type of Gloss	Characteristic Appearance	Glossiness Range in Which Prevalent
Specular gloss	Brilliance of high lights, shininess	Medium glossiness
Sheen	Shininess at grazing angles	Low glossiness
Contrast gloss	Contrast between specularly reflecting areas and other areas	Low glossiness
Distinctness-of-image gloss	Distinctness and sharpness of mirror images	High glossiness
Absence-of-bloom gloss	Absence of smear or excess semi-specular reflection adjacent to high lights	High glossiness
Surface-uniformity gloss	Surface uniformity, freedom from visible nonuniformities	Medium to high glossiness

Tentative Method of Test for

DAYLIGHT 45 DEGREE, 0 DEGREE APPARENT REFLECTANCE OF PAINT FINISHES[1]

A.S.T.M. Designation: D 771 - 44 T

ISSUED, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. This method of test covers a procedure for daylight 45 deg., 0 deg. apparent reflectance of paint finishes. The combination of illumination at 45 deg. with perpendicular viewing as prescribed in this method is in accord with the recommendation of the International Commission on Illumination as the standard condition for the colorimetry of opaque surfaces[3] because it represents a satisfactory average of the directional conditions under which the nonglossy attributes of surface colors are observed in everyday life. Values of daylight 45 deg., 0 deg. apparent reflectance are therefore useful and are widely used for designating the approximate daylight diffuse reflectances of opaque surfaces and for indicating the lightnesses which these surfaces will appear to have.

NOTE.—Values of luminous apparent reflectance for nearly perpendicular illumination and nearly completely diffuse viewing are also employed for the same two purposes. Two geometries of this sort are employed by the General Electric recording photoelectric spectrophotometer and by other reflectometers; in one geometry, the viewing fails to be completely diffuse chiefly because some light is lost in the direction of the illuminating beam, in the other geometry, light reflected along any of a group of angles surrounding the direction of specular reflection is purposely kept from the receptor. With surfaces which are both of light color (45 deg., 0 deg. luminous apparent reflectance more than 50 per cent) and nearly or quite devoid of gloss, values of apparent reflectance for the 45 deg., 0 deg. and 0 deg. diffuse geometries are nearly identical. For surfaces which are either somewhat glossy or dark colored, values of 0 deg. diffuse luminous apparent reflectance are generally higher than values of 45 deg., 0 deg. luminous apparent reflectance. With dark surfaces in particular, the inclusion of light reflected along or near the direction of specular reflection in measurements by instruments employing the 0 deg. diffuse geometry causes these instruments to give values of apparent reflectance that are too high to accord with lightness ratings made visually.

Definition

2. *Daylight 45 deg., 0 deg. Apparent Reflectance.*—The daylight 45 deg., 0 deg. apparent reflectance of a surface is de-

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Accepted by the Society at annual meeting, June, 1944.

[3] *Proceedings*, Eighth Session, Commission Internationale de l'Eclairage, Cambridge, England, September, 1931, pp. 19–29. See also Standard Method of Test for Spectral Characteristics and Color of Objects and Materials (A.S.T.M. Designation: D 307), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

fined as the daylight reflectance an ideally diffusing specimen would have to have to appear as bright as the test specimen when both are illuminated at 45 deg. by I.C.I. illuminant *C*[3] (daylight) and viewed perpendicularly by a normal observer possessing the I.C.I. luminosity function.[3]

Apparatus

3. (*a*) *Spectral Characteristics.*—The source shall approximate standard illuminant *C* (representative of average daylight) recommended by the International Commission on Illumination (I.C.I.),[3] and the receptor shall have a spectral sensitivity approximating the luminosity function of the standard I.C.I. observer[3] or equivalent source-receptor combinations may be used.

(*b*) *Geometric Characteristics.*—Illumination shall be within 4 deg. of, and centered about, a direction 45 deg. from the perpendicular to the test surface; viewing shall be within 15 deg. of, and centered about, the perpendicular. The above directions of illuminating and viewing may be interchanged giving the equivalent 0 deg., 45 deg. conditions.

NOTE.—The Hunter multipurpose reflectometer, used with green filter in the manner described by Richard S. Hunter, "A Multipurpose Photoelectric Reflectometer," *Research Paper RP 1345*, Nat. Bureau Standards, November, 1940, meets the requirements prescribed in Section 3.

Standards

4. (*a*) *Primary Standard.*—The primary standard is the ideal, completely reflecting, perfectly diffusing surface. Experiment has shown that a freshly smoked surface of magnesium oxide prepared according to the instructions in National Bureau of Standards *Letter Circular LC 547* (1939) is not significantly different from the primary standard in its power to reflect light perpendicularly when illuminated at 45 deg. (*Warning*: Surfaces of magnesium carbonate or aged surfaces of MgO may be significantly lower in apparent reflectance than freshly prepared MgO).

(*b*) *Working Standards.*—Porcelain-enameled metal plaques or other materials known to be reasonably permanent in their reflective properties can be calibrated and used as working standards of daylight 45 deg., 0 deg. apparent reflectance. For each measurement, it is recommended that the available working standard closest in daylight 45 deg., 0 deg. apparent reflectance to the sample be chosen.

Preparation of Sample

5. For the daylight 45 deg., 0 deg. apparent reflectance of a given system of coating-on-ground, instructions for the preparation of the system shall be followed. For the daylight 45 deg., 0 deg. apparent reflectivity of a paint material, sufficient coats of the material shall be applied to a white vitreous-enamel, sized cardboard, or other suitable panel until an additional coat produces no measurable change in the reflectance reading. In either case the last coat shall be allowed to dry thoroughly in a well-lighted room or cabinet free from dust or fumes.

Report

6. Unless otherwise specified, values of daylight 45 deg., 0 deg. apparent reflectance shall be reported to the nearest 1 per cent.

Tentative Definitions of

TERMS RELATING TO PAINT, VARNISH, LACQUER, AND RELATED PRODUCTS[1]

A.S.T.M. Designation: D 16 - 44 T

ISSUED, 1940; REVISED, 1941, 1942, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Alkyd Resin.—Under Resin, see Alkyd Resin.

Baking Finish.—A paint or varnish that requires baking at temperatures above 150 F. for the development of desired properties.

Baking Temperature.—A temperature above 150 F. (65.6 C.).

Bituminous Varnish—Under Varnish, see Bituminous Varnish.

Bleeding.—The diffusion of coloring matter through a coating from the substrate; also, the discoloration arising from such diffusion.

Bulking Value.—Solid volume of a unit weight of material usually expressed as gallons per pound.

Color.—The aspect of the appearance of objects which depends upon the spectral composition of the light reaching the retina of the eye and upon its temporal and spatial distribution. Black, white, and gray are colors as well as red, yellow, green, blue, purple, and their intermediates. Mixtures or blends of these are also colors. The colors of objects have three attributes: hue, lightness and saturation.

NOTE—Appearance depends upon shape, gloss, transparency, and texture as well as upon color. The term *color* is sometimes loosely used in reference to material substances (pigments, stains, dyes) apart from their appearance.

Hue.—The attribute which determines whether the color is red, yellow, green, blue, purple, or the like.

Lightness.—The attribute which permits an object color to be classified as equivalent to some member of the series of grays ranging between black and white.

Saturation.—The attribute of any color possessing a hue which determines the degree of its difference from the gray of the same lightness.

Drier.—A composition which accelerates the drying of oil, paint, or varnish. Driers are usually metallic compositions and are available in both solid and liquid forms.

Drying Oil.—An oil which possesses to a marked degree the property of readily

[1] Under the standardization procedure of the Society, these definitions are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Prior to their present publication as tentative these definitions were published as proposed standard definitions from 1914 to 1915, being revised in 1915. They were adopted in 1915, published as standard from 1915 to 1940, being revised in 1922 and 1924, but revised and republished as tentative in 1940.

taking up oxygen from the air and changing to a relatively hard, tough, elastic substance when exposed in a thin film to the air.

Enamel.—A paint which is characterized by an ability to form an especially smooth film.

Filler.—A pigmented composition for filling the pores or irregularities in a surface preparatory to application of other finishes.

Flatting Agent.—A material added to paints, varnishes, and other coating materials to reduce the gloss of the dried film.

Forced Drying Temperature.—A temperature between room temperature and 150 F. (65.6 C.).

Fossil Resin.—Under Resin, see Fossil Resin.

Glaze.—A very thin coating of a paint product usually a semi-transparent coating tinted with Van Dyke brown, burnt sienna, or a similar pigment; applied on a previously painted surface to produce a decorative effect.

Hiding Power.—The power of a paint or paint material as used to obscure a surface painted with it.

In this definition the word "obscure" means to render invisible or to cover up a surface so that it cannot be seen.

Hue.—Under Color, see Hue.

Lacquer.—A liquid composition containing cellulose esters or ethers as the basic film-forming ingredients. Lacquer dries rapidly by solvent evaporation.

Sanitary or Tin Plate Lacquers.—Usually varnish coatings for application on the interior of metal food containers.

Lake.—A special type of pigment consisting essentially of an organic soluble coloring matter combined more or less definitely with an inorganic base or carrier. It is characterized generally by a bright color and a more or less pronounced translucency when made into an oil paint.

Under this term are included two (and perhaps three) types of pigment: (*a*) the older original type composed of hydrate of alumina dyed with a solution of the natural organic color, (*b*) the more modern and far more extensive type made by precipitating from solution various coal-tar colors by means of a metallic salt, tannin, or other suitable reagent, upon a base or carrier either previously prepared or coincidently formed, and (*c*) a number combining both types in varying degree, might be regarded as a third class.

Lightness.—Under Color, see Lightness.

Natural Resin.—Under Resin, see Natural Resin.

Nondrying Oil.—An oil which does not of itself possess to a perceptible degree the power to take up oxygen from the air and lose its liquid characteristics.

Nonvolatile Vehicle.—The liquid portion of a paint excepting its volatile thinner and water.

Oil Paint.—Under Paint, see Oil Paint.

Oil Varnish.—Under Varnish, see Oil Varnish.

Opacity.—The degree of obstruction to the transmission of visible light.

In this sense "opacity" is a relative term it being considered that given a film sufficiently thin, in paint technology at least, there is no absolutely opaque substance.

Paint.—A pigmented liquid composition which is converted to an opaque solid film after application as a thin layer.

Oil Paint.—A paint that contains drying oil or oil varnish as the basic vehicle ingredient.

Water Paint.—A paint that contains a water emulsion or dispersion as the vehicle.

Paste Paint.—A paint in which the pigment is sufficiently concentrated to permit a substantial reduction with vehicle before use.

Paste Paint.—Under Paint, see Paste Paint.

Phenolic Resin.—Under Resin, see Phenolic Resin.

Pigment.—The fine solid particles used in the preparation of paint, and substantially insoluble in the vehicle.

Asphaltic materials are not pigments except when they contain substances substantially insoluble in the vehicle in which they are used.

Pigment Volume.—The percentage by volume of pigment in the nonvolatile portion of a paint as calculated from bulking value and composition data. The letters PV are commonly used as an abbreviation.

Putty.—A stiff, dough-like material consisting of pigment and vehicle, used for setting window panes and filling imperfections in wood or metal surfaces.

Resin.—A solid or semisolid, water-insoluble, organic substance with little or no tendency to crystallize.

Natural Resin.—A resin formed by natural processes.

Fossil Resin.—A natural resin of ancient origin usually found in the earth.

Synthetic Resin.—A resin manufactured by chemical processes.

Alkyd Resin.—A synthetic resin made from polyhydric alcohols and polybasic acids.

Phenolic Resin.—A synthetic resin made from phenols and aldehydes.

Sanitary or Tin Plate Lacquers.—Under Lacquer, see Sanitary or Tin Plate Lacquers.

Saturation.—Under Color, see Saturation.

Semidrying Oil.—An oil which possesses the characteristics of a drying oil but to a lesser degree.

There is no definite line of demarcation between drying and semidrying oils.

Shade.—A term descriptive of a lightness difference between surface colors, the other attributes of color being essentially constant. A lighter shade of a color is one that has higher lightness but approximately the same hue and saturation; and a darker shade is one that has a lower lightness.

Primarily, the term "shade" is derived from shadow and designates a change in appearance analogous to that produced by a local reduction in illumination. It should, therefore, when strictly used, express only the change toward a darker color. Shade of a color has been defined by several authorities as the mixture of black with that color, thus establishing its opposite character to "tint", but by extension of its relative sense it has been frequently and widely used to include lighter shades by use of the adjective "lighter" or "paler". Although such expressions apparently involve a contradiction, it is clear that while we may have a shade of a color, or darker color of the same sort, it is easy to conceive of another shade not quite so dark and therefore lighter.

Size.—A liquid coating composition, usually transparent, for sealing a porous surface preparatory to application of finishing coats.

Spirit Varnish.—Under Varnish, see Spirit Varnish.

Spreading Rate.—The area covered by a unit volume of coating material frequently expressed as square feet per gallon.

Surfacer.—A pigmented composition for filling minor irregularities to obtain a smooth uniform surface preparatory to applying finish coats; usually applied over a primer and sandpapered for smoothness.

Synthetic Resin.—Under Resin, see Synthetic Resin.

Tint.—A color produced by the mixture of white pigment or paint in predominating amount with a colored pigment or paint, not white. The tint of a color is, therefore, much lighter and much less saturated than the color itself.

Tinting Strength.—The power of coloring a given quantity of paint or pigment selected as a medium standard for estimating such power.

Toner.—An organic pigment which does not contain inorganic pigment or inorganic carrying base.

Varnish.—A liquid composition which is converted to a transparent or translucent solid film after application as a thin layer.

Bituminous Varnish.—A dark colored varnish containing bituminous ingredients. The varnish may be either of the oil or spirit type.

Oil Varnish.—A varnish which contains resin and drying oil as the basic film forming ingredients and is converted to a solid film primarily by chemical reaction.

Spirit Varnish.—A varnish which is converted to a solid film primarily by solvent evaporation.

Vehicle.—The liquid portion of a paint.

Anything that is dissolved in the liquid portion of a paint is a part of the vehicle.

Volatile Thinner.—All that liquid portion of a paint, water excepted, which is volatile in a current of steam at atmospheric pressure.

Water Paint.—Under Paint, see Water Paint.

Tentative Specifications for

WOOD TO BE USED AS PANELS IN WEATHERING TESTS OF PAINTS AND VARNISHES[1]

A.S.T.M. Designation: D 358 – 44 T

ISSUED, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Purpose and Scope

1. These specifications designate woods for weathering tests on exterior oil paints and other materials of similar purpose. Such tests may include either outdoor exposure tests or accelerated laboratory tests. According to experience, the wood used as a test surface has a marked bearing upon weathering results, especially in accelerated laboratory tests or 45-deg. outdoor exposures. It is the purpose of these specifications to maintain more uniformity of the influence of the wood, particularly in cooperative work among different laboratories. The cedar and white pine, as specified, are desirable for determining general durability factors. Cedar, as specified, is affected less by the exposure conditions and is preferred for accelerated tests (laboratory and 45-deg. outdoor exposures) to avoid the masking of the true failure characteristics of the coating by unusual changes in the wood surface. Redwood, as specified, may be substituted for western red cedar when such substitution is mutually agreed upon or when the specified grade of western red cedar is not available. From the standpoint of being affected by exposure conditions, white pine, as specified, represents an intermediate wood. Southern yellow pine, as specified, emphasizes the influence of wood on adhesion and durability of the coating.

WESTERN RED CEDAR

Species

2. The material for test panels shall be western red cedar (*Thuja plicata*).

Weight per Cubic Foot

3. The wood shall weigh 22 to 23 lb. per cu. ft. The weight shall be determined after at least one week's storage in a protected, dry, warm atmosphere (approximately 70 F. (21 C.)).

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Accepted by Committee E-10 on Standards, August 28, 1944.

Prior to their present publication as tentative, these specifications were published as tentative from 1933 to 1938, being revised in 1935. They were adopted in 1938, published as standard from 1938 to 1944, but were withdrawn, revised, and republished as tentative in 1944.

Character of Wood

4. The material shall be close grained, as nearly edge grain as possible and in no section to vary more than 45 deg. from edge grain. The surfaces shall be smoothly planed and sanded.

Thickness of Panels

5. The minimum thicknesses (Note) in proportion to width (across the grain) shall be as follows:

Panel Width, in.	Minimum Thickness, in.
3 and under	$\frac{3}{8}$
Over 3 to 6, incl	$\frac{7}{16}$
Over 6	$\frac{1}{2}$

NOTE.—Minimum thicknesses in proportion to width (across the grain) do not apply to panels made of clapboard or siding of commercial types, which shall be used as regularly marketed.

WHITE PINE

Species

6. The white pine wood shall be restricted to northern white pine (*Pinus strobus*) and Idaho white pine (*Pinus monticola*).

Weight per Cubic Foot

7. The wood shall weight 24 to 26 lb. per cu. ft. when wood is in equilibrium with air at 60 to 65 per cent relative humidity and 69 to 71 F. (20.5 to 21.6 C.) (wood at approximately 12 per cent moisture content).

Character of Wood

8. (*a*) The wood shall be free from knots, pitch pockets, pitch streaks, and other defects. The number of annual growth rings per inch along the radius of the log from which obtained shall be not less than 18. The surfaces shall be smoothly planed and sanded.

(*b*) The wood shall be selected as edge grain or flat grain as desired. Specimens in which the annual growth rings intersect the planed test surface at an angle of 45 deg. or more shall be considered edge grain, and similarly specimens in which the intersection is at an angle of 15 deg. or less shall be considered as flat grain. The test surface of flat grain wood shall be on the bark side.

Thickness of Panels

9. The minimum thicknesses (see Note, Section 5) in proportion to width (across the grain) shall be as follows:

Panel Width, in.	Minimum Thickness, in.
3 and under	$\frac{7}{16}$
Over 3 to 6, incl	$\frac{1}{2}$
Over 6	$\frac{5}{8}$

SOUTHERN YELLOW PINE

Species

10. The material for test panels shall be southern yellow pine, suitable for siding purposes. It is not to be limited to a single species, but shall conform to the requirements prescribed in Sections 11 and 12.

Weight per Cubic Foot

11. The wood shall weigh 32 to 38 lb. per cu. ft. when wood is in equilibrium with air at 60 to 65 per cent relative humidity and 69 to 71 F. (20.5 to 21.6 C.) (wood at approximately 12 per cent moisture content).

Character of Wood

12. (*a*) The wood shall be free from knots, pitch pockets, pitch streaks, and other defects. The number of annual growth rings per inch along the radius of the log from which obtained shall be not less than 7 nor more than 9. The surfaces shall be smoothly planed and sanded.

(*b*) The wood shall be selected as flat grain; specimens in which the annual growth rings intersect the planed test surface at an angle of 15 deg. or less shall be considered as flat grain.

Thickness of Panels

13. The minimum thicknesses (see Note, Section 5) in proportion to width (across the grain) shall be as follows:

Panel Width, in.	Minimum Thickness, in.
3 and under	$\frac{7}{16}$
Over 3 to 6, incl.	$\frac{3}{4}$
Over 6	$\frac{7}{8}$

REDWOOD

Species

14. The material for test panels shall be redwood (*Sequoia sempervirens*).

Weight per Cubic Foot

15. The wood shall weigh 28 to 29 lb. per cu. ft. at 12 per cent moisture content. The weight shall be determined after at least one week's storage in a protected, dry, warm atmosphere (approximately 70 F. (21 C.)).

Character of Wood

16. The material shall be close grained, as nearly edge grain as possible, and in no section to vary more than 45 deg. from edge grain. The number of annual growth rings per inch along the radius of the log from which obtained shall be not less than 18. The wood shall be free of knots, oil streaks, and other defects such as wavy burl grain. The surfaces shall be smoothly planed and sanded.

Thickness of Panels

17. The minimum thicknesses (see Note, Section 5) in proportion to width (across the grain) shall be as follows:

Panel Width, in.	Minimum Thickness, in.
3 and under	$\frac{3}{8}$
Over 3 to 6, incl.	$\frac{1}{2}$
Over 6	$\frac{3}{4}$

Tentative Method for

PREPARATION OF STEEL PANELS FOR EXPOSURE TESTS OF ENAMELS FOR EXTERIOR SERVICE[1]

A.S.T.M. Designation: D 609 - 41 T

ISSUED, 1941.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. This method covers two procedures for the preparation of steel panels for exposure tests of enamels for exterior service.

Test Panels

2. The test panels shall be prepared from new cold-rolled steel, No. 20 to No. 22 gage, that has been oiled at the mill and is free of rust. The steel shall conform to the requirements of grade No. 5, dead soft temper, of the Standard Specifications for Cold-Rolled Strip Steel (A.S.T.M. Designation: A 109) of the American Society for Testing Materials,[3] and shall have a dull finish. The panels shall be not less than 2½ by 5 in. in size.

Preparation of Panel Surface

3. The test panels shall be prepared for painting by one of the following procedures:

Procedure A.—The test panels, free of rust, shall be cleaned with suitable grease-removing solvents such as benzol, toluol, naphtha, trichlorethylene vapors, or water without inhibitors or soaps, so that the surface of the panel shows no water break when immersed in distilled water. The panel shall then be immersed in alcohol (absolute ethyl, isopropyl, or suitable denatured alcohol), removed and allowed to dry, and finished, or stored in a desiccator until ready for finishing.

Procedure B.—The panels, free of rust, shall be cleaned of all grease and oil by suitable solvents such as benzol, toluol, naphtha, or trichlorethylene vapors, and subjected to a phosphate solution until a uniform phosphate coating is obtained, the conditions of treatment to be agreed upon by the purchaser and the seller.

Application of Finishing Material

4. The finishing material shall be applied by a method agreed upon by the purchaser and the seller. The number of coats, the thickness of each coat, the time and manner of drying each coat, as well as the total thickness of the finish shall also be agreed upon by the purchaser and the seller. Each individual coat shall be applied to a uniform dry film thickness over any panel or group of panels within plus or minus 0.0002 in. of the specified thickness.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Accepted by the Society at annual meeting, June, 1941.

[3] 1944 Book of A.S.T.M. Standards, Part I.

Tentative Method for

EVALUATING THE DEGREE OF RESISTANCE TO BLISTERING OF ORGANIC COATINGS ON METAL WHEN SUBJECTED TO IMMERSION OR OTHER TESTS INVOLVING EXPOSURE TO MOISTURE OR LIQUIDS[1]

A.S.T.M. Designation: D 714 - 43 T

ISSUED, 1943.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. The photographic reference standards included in this method are representative of different sizes of blisters that may develop on paint systems on iron, steel, or other metal surfaces when subjected to immersion tests (Note). They are to be used for comparative purposes and are not intended to have a direct relationship to a decision regarding what constitutes failure of the paint system. This shall be agreed upon by the purchaser and seller.

NOTE.—These photographic standards were obtained by immersion of paint systems on steel panels in tap water but, for descriptive purposes, they may be useful in other tests involving exposure to moisture or liquids.

Types of Blistering

2. The blistering represented in these standards ranges from the small "pin point" type through the intermediate sizes to the large and more or less irregular "globular" types.

Use of Photographic Reference Standards

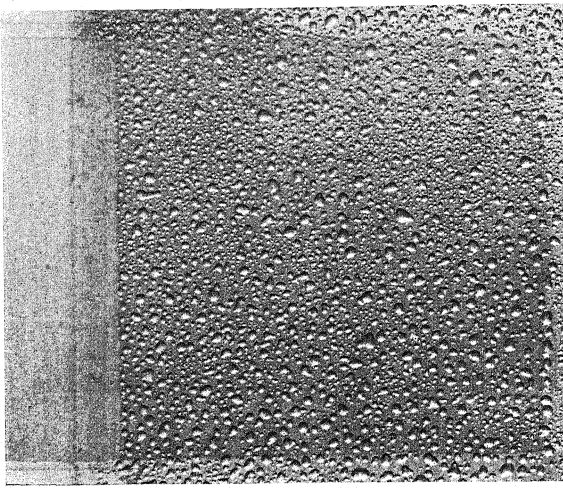
3. The use of the photographic reference standards shown in Figs. 1 and 2 is subject to the following interpretations and suggestions:

(*a*) The photographic reference standards are intended to designate sizes of blisters and not quantity or distribution of blistering.

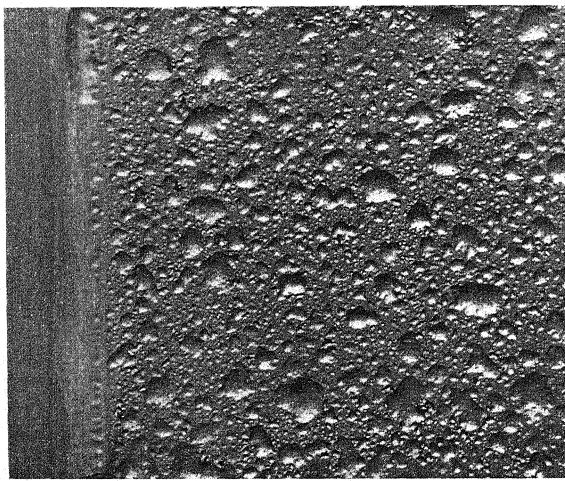
(*b*) The selection of reference standards has been restricted to four steps within a scale of 0 to 10, in which 0 represents the presence of very large blisters and 10 represents no blistering under the conditions of the test. These steps are designated as Blistering Standards No. 2, No. 4, No. 6, and No. 8, respectively. Intermediate stages shall be judged by interpolation. The number shall refer to the larger sized blisters on the respective photographic standards (Note 1).

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Accepted by the Society at annual meeting, June, 1943.

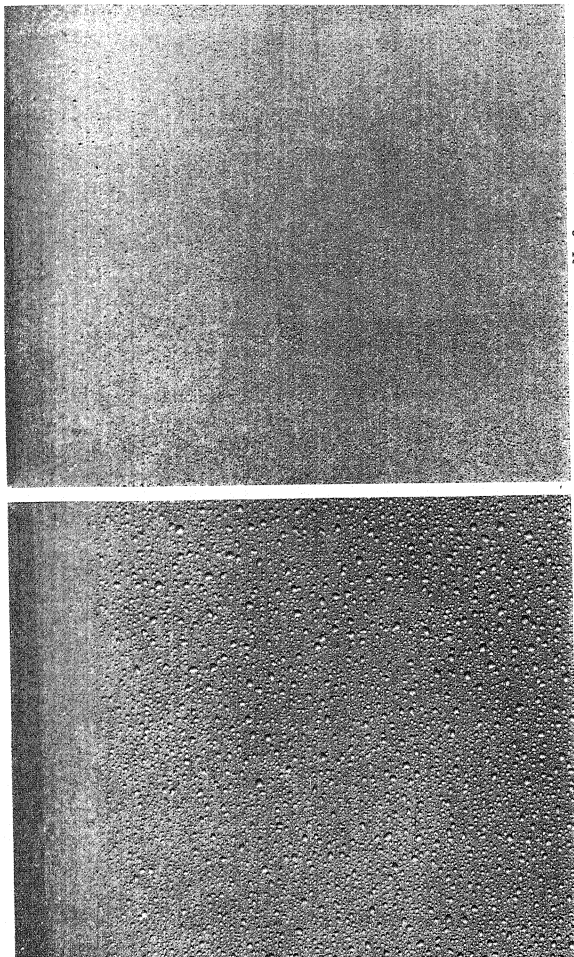


No. 4.



No. 2.

FIG. 1.—Degrees of Blistering.



No. 8. No. 6.

FIG. 2.—Degrees of Blistering.

NOTE 1: *Example.*—Photographic standard No. 4 has on it blisters ranging in size from about No. 7 to No. 4, inclusive. This is necessary because it appears impractical to obtain photographic reference standards that show only one size of blistering uniformly distributed.

(*c*) Blistering shall be reported in terms of amount or percentage of the area on the test specimen that is covered by blisters of different sizes (Note 2) as defined and illustrated by the respective reference standards and as specified in Paragraph (*b*).

NOTE 2: *Example.*—The test specimen shows No....to No....blistering on...per cent of the surface.

(*d*) When the amount and size of blistering vary considerably on different parts of the test specimen, this shall be indicated by reporting, in so far as practical, the extent of this variation by statements such as:

No.....to No.....blistering on....per cent of the surface.
No.....to No.....blistering on....per cent of the surface, etc.

(*e*) While reference to one or more of the standard numbers conveys the idea of "uniform" or "nonuniform" size, additional descriptive words such as "concentrated" or "scattered" and "general" or "localized" are needed and should be used (Note 3).

NOTE 3: *Example.*—Standard No. 4 would be described as follows:
No. 7 to No. 4 concentrated blistering general on 100 per cent of the surface.

Tentative Method of

EVALUATING DEGREE OF RESISTANCE TO FLAKING (SCALING) OF EXTERIOR PAINTS OF THE LINSEED-OIL TYPE[1]

A.S.T.M. Designation: D 772 - 44 T

ISSUED, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. The photographic reference standards included in this method are representative of degrees of resistance to flaking (scaling) of linseed-oil-type exterior paint films on wood surfaces. These standards are primarily intended for comparative evaluation.

Definition

2. Flaking (scaling) is that phenomenon manifested in paint films by the actual detachment of pieces of the film itself either from its substrate or from paint previously applied. Flaking (scaling) is generally preceded by cracking or checking and is the result of loss of adhesion, usually due to stress-strain factors coming into play.

Types of Flaking (Scaling)

3. Only one type of flaking (scaling) is recognized, as defined in Section 2.

Use of Photographic Reference Standards

4. The use of the photographic reference standards[3] shown in Fig. 1 requires the following precautions:

(*a*) Care must be taken not to confuse various types of failure that may be present on the same surface.

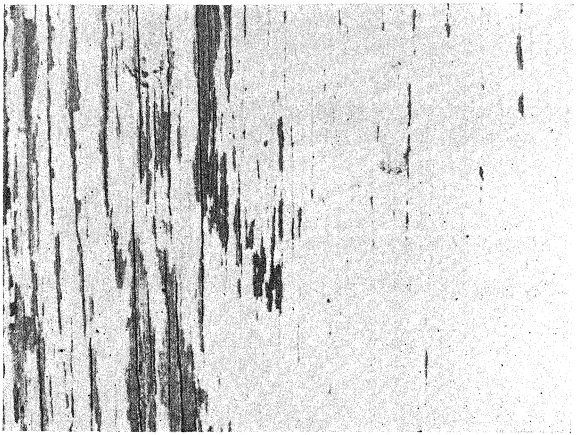
(*b*) It must be realized that degree of failure will vary over any given area. Therefore, an average portion of the film should be used for comparison.

(*c*) In technical literature, a distinction is sometimes made between flaking and scaling. In most cases, however, flaking and scaling refer to the same phenomenon. In some instances, the term flaking is used to describe the detachment of pieces of film less than $\frac{1}{4}$ in. in size, and scaling, the detachment of pieces over $\frac{1}{4}$ in. in size. In other instances, the term flaking is used to describe the detachment of pieces of film

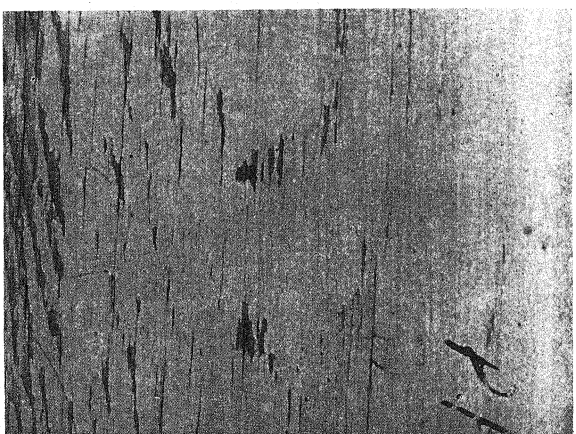
[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

[2] Accepted by the Society at annual meeting, June, 1944.

[3] Copies of the Exposure Standards Manual prepared by the Federation of Paint and Varnish Production Clubs, giving actual photographs of various types of failures of exterior paints, may be obtained from the Secretary of the Federation, 704 Weightman Building, 1524 Chestnut St., Philadelphia 2, Pa.

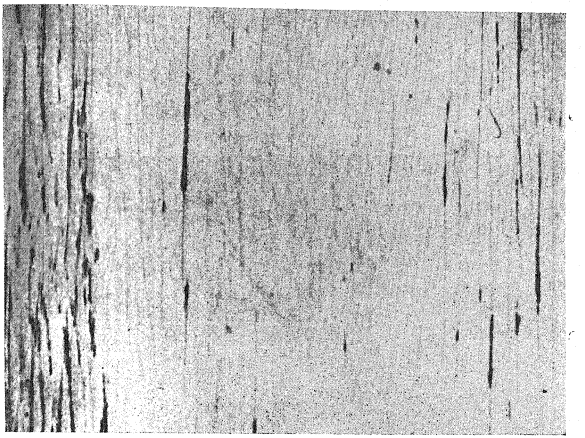


No. 2

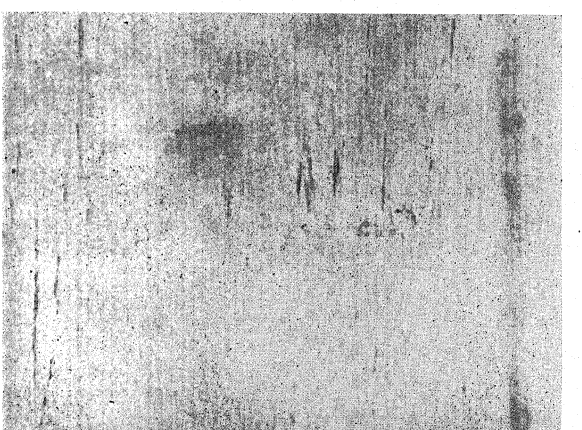


No. 4

FIG. 1. — Degrees of Flaking (Scaling).



No. 6



No. 8

FIG. 1. — Degrees of Flaking (Scaling) (*Concluded*).

from the immediate undercoat (intercoat failure) and scaling the detachment of pieces from the base (complete failure). It should be kept in mind that the flakes may vary widely in size and shape from those illustrated by the reference standards in Fig 1, varying from a fraction of an inch to several inches in size.

(*d*) Peeling should be considered as an aggravated form of flaking (scaling) and not as a different type of failure. It is frequently due to a moisture condition and when this is evident it should be taken into consideration in any evaluation.

(*e*) For convenience in recording data obtained, the record sheet approved by the Federation of Paint and Varnish Production Clubs is recommended.[4]

[4] These record sheets may be obtained from the Palmerton Printing Co., Palmerton, Pa., or from the American Society for Testing Materials, 260 S. Broad St., Philadelphia 2, Pa.

Tentative Specifications for

A.S.T.M. THERMOMETERS[1]

A.S.T.M. Designation: E 1 - 44 T

ISSUED, 1939; REVISED, 1940, 1941, 1942, 1943, 1944.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These specifications cover glass thermometers graduated in Centigrade or Fahrenheit degrees and frequently specified in methods of the American Society for Testing Materials. The various thermometers covered are listed in Table I.

Requirements

2. The individual thermometers shall conform to the detailed specifications given in Table I and to the general requirements specified in Sections 3 to 11.

NOTE.—For the purpose of interpreting these specifications the following descriptions of terms apply:

The total length is the over-all length of the finished instrument.

The diameter is that measured with a ring gage or micrometer unless otherwise specified in Table I.

The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing.

The top of the thermometer is the top of the finished instrument.

Type

3. Each thermometer shall be of the etched glass stem type unless otherwise specified in Table I.

Stem

4. The stem shall be made of suitable thermometer tubing and shall have a plain front and enamel back unless otherwise specified in Table I.

Bulb

5. The bulb shall be made of Corning normal or equally suitable thermometric glass.

Graduations

6. All graduation lines, figures, and letters shall be clear-cut and distinct.

Special Marking

7. In addition to the special markings prescribed in Table I, each thermometer shall be marked with a serial number and the manufacturer's name or trade mark etched on the stem.

[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee E-1 on Methods of Testing.

[2] Latest revisions accepted by Committee E-10 on Standards, March 15, 1944, and by the Society at annual meeting, June, 1944.

These specifications when adopted as standard are intended to be incorporated in the Standard Specifications for A.S.T.M. Thermometers (E 1 - 44), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Scale Error

8. The error at any point of the scale up to the temperature limit prescribed in Table I when the thermometer is standardized in accordance with Section 9 shall not exceed the values prescribed in Table I.

Standardization

9. The thermometer shall be standardized immersed in the testing bath to the top of the mercury column, unless other conditions of immersion are prescribed in Table I, and at the temperatures prescribed in Table I. (See Explanatory Notes.)

Test for Permanency of Range

10. The test for permanency of range shall be made at the temperature prescribed in Table I and under the immersion conditions specified for the thermometer. The accuracy after the test shall be within the limit specified.

Case

11. The thermometer shall be supplied in a suitable case on which shall appear the marking prescribed in Table I.

EXPLANATORY NOTE ON PERIODIC CHECKING OF CHANGES OF BULB VOLUME OF GLASS THERMOMETERS

A mercury in glass thermometer, if properly made and used, is an extremely reliable instrument but small changes in volume of the thermometer bulb may occur, even though the thermometer may have been carefully annealed or aged.

Usually the bulb contracts slowly and as a result the thermometer may read higher after it has been placed in service than it did originally. These changes are more appreciable for short-range thermometers graduated in fractional degrees, and the users of such thermometers should check such instruments from time to time. When first received the thermometers should be checked every week or so, later these time intervals may be lengthened if the changes are not of consequence.

The ice-point method (reading in melting ice at 0 C. or 32 F.) has been found to be the most convenient and at the same time the most accurate method for checking changes in bulb volume. Other temperatures, such as the boiling point of water and of other pure substances, have been proposed for "fixed points," but have not been found entirely satisfactory owing to the difficulties of accurately obtaining these fixed temperatures.

Short-range thermometers graduated in fractional degrees may or may not be provided with an ice point reading if this temperature is not included in the given range. This extra point can be incorporated in the thermometer, but may be omitted for the sake of economy. In the case of distillation thermometers intended to be inserted in the neck of a flask, the ice-point graduation may be undesirable since the contraction chamber between the ice point and the first graduation may contain enough mercury to cause uncertain and variable readings. For this reason the ice-point graduation, unless it is an integral part of the range, is generally omitted from the specifications for distillation thermometers.

In the absence of the ice point other means must be provided for detecting changes in the readings, especially if the thermometer is fractionally graduated. The method most commonly used involves the comparison at one or more temperatures in a well-stirred, properly constructed liquid bath of the thermometer to be checked against another standardized thermometer of similar specifications to the test thermometer but provided with an ice point.

Where several thermometers make up a series or set for some specific method and these thermometers do not have ice points, a similar set may be provided with ice points, and these should be kept for use as reference standards for checking routine test thermometers.

Detailed instructions for making such comparisons and specifications for suitable testing apparatus, as well as other data on this subject, will be found in the paper by E. F. Mueller and R. M. Wilhelm, "Methods of Testing Thermometers," *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part I, p. 493 (1938).

EXPLANATORY NOTE ON ICE POINT DETERMINATIONS OF GLASS THERMOMETERS

Two methods have been in use for determining the ice point on thermometers graduated fractionally and with an open scale, such as those of the Kinematic type. One method requires that the thermometer remain at approximately room temperature for 72 hr. before the ice point is taken. The other stipulates that the ice point reading be taken immediately or within a definitely stated short period after heating to the test temperature. Changes in the correction of the thermometer may be observed by determining the changes in the ice point by either method.

Since it may not be practical to wait until the thermometer has rested for three or more days, the method as outlined below is suggested. These instructions are given in the form of a note which should appear in the table of corrections for the thermometer.

NOTE.—The tabulated corrections apply for the condition of immersion indicated provided the ice point reading taken after heating to ····· for not less than three minutes is ····· .

If the ice point reading (taken in not less than 2 min. and not more than 5 min. after removal of the thermometer from the heated bath) is found to be higher (or lower) than stated, all other readings will be higher (or lower) to the same extent.

(See Table I, pp. 1570 to 1574)

TABLE I.—REQUIREMENTS FOR A.S.T.M. THERMOMETERS.

Name	A.S.T.M. Kinematic Viscosity[a]	A.S.T.M. Kinematic Viscosity[a]	A.S.T.M. Kinematic Viscosity[a]
A.S.T.M. Designation	E 1 (28F – 43T)	E 1 (29F – 43T)	E 1 (30F – 43T)
Liquid	mercury	mercury	mercury
Filling above Liquid	nitrogen gas	nitrogen gas	nitrogen gas
Temperature Range For Tests at	97.5 to 102.5 F. 100 F.	127.5 to 132.5 F. 130 F.	207.5 to 212.5 F. 210 F.
Subdivisions	0.1 F.	0.1 F.	0.1 F.
Total Length	240 to 245 mm. (9.45 to 9.65 in.)	240 to 245 mm. (9.45 to 9.65 in.)	240 to 245 mm. (9.45 to 9.65 in.)
Stem Diameter	6.5 to 7.5 mm. (0.256 to 0.295 in.)	6.5 to 7.5 mm. (0.256 to 0.295 in.)	6.5 to 7.5 mm. (0.256 to 0.295 in.)
Bulb Diameter	6.0 mm. (0.236 in.) to not greater than stem	6.0 mm. (0.236 in.) to not greater than stem	6.0 mm. (0.236 in.) to not greater than stem
Bulb Length	45 to 55 mm. (1.77 to 2.17 in.)	45 to 55 mm. (1.77 to 2.17 in.)	45 to 55 mm. (1.77 to 2.17 in.)
Ice Point: Range graduated	31.5 to 32.5 F.	31.5 to 32.5 F.	31.5 to 32.5 F.
Ice Point: Subdivisions	0.1 F.	0.1 F.	0.1 F.
Ice Point: Bottom of bulb to 32 F	77 to 87 mm. (3.03 to 3.43 in.)	77 to 87 mm. (3.03 to 3.43 in.)	77 to 87 mm. (3.03 to 3.43 in.)
Contraction Chamber: Bottom of chamber to bottom of bulb, min	100 mm. (3.94 in.)	100 mm. (3.94 in.)	100 mm. (3.94 in.)
Contraction Chamber: Top of chamber to bottom of bulb, max	125 mm. (4.92 in.)	125 mm. (4.92 in.)	125 mm. (4.92 in.)
Expansion Chamber	permit heating to 220 F.	permit heating to 220 F.	permit heating to 220 F.
Bottom of Bulb to Graduation Line at Distance	97.5 F. 137 to 157 mm. (5.39 to 6.18 in.)	127.5 F. 137 to 157 mm. (5.39 to 6.18 in.)	207.5 F. 137 to 157 mm. (5.39 to 6.18 in.)
Top of Thermometer to Graduation Line at Distance	102.5 F. 27 to 47 mm. (1.06 to 1.85 in.)	132.5 F. 27 to 47 mm. (1.06 to 1.85 in.)	212.5 F. 27 to 47 mm. (1.06 to 1.85 in.)
Top Finish	glass ring	glass ring	glass ring
Longer Graduation Lines at Each	0.5 F.	0.5 F.	0.5 F.
Graduations Numbered at Each Multiple of	1 F.	1 F.	1 F.
Immersion	total	total	total
Special Marking on Thermometer (See Section 7)	ASTM Kin Vis	ASTM Kin Vis	ASTM Kin Vis
Scale Error at when standardized, shall not exceed	100 F. 0.10 F.	130 F. 0.10 F.	210 F. 0.10 F.
Standardization	The thermometers shall be standardized at the ice point,[b] and at the viscosity test point (100, 130, or 210 F.) for conditions of total immersion, expressed to the nearest 0.02 F. and such corrections shall be added to the observed reading.		
Marking on Case	A.S.T.M. Kinematic Viscosity Thermometer 97.5 to 102.5 F.	A.S.T.M. Kinematic Viscosity Thermometer 127.5 to 132.5 F.	A.S.T.M. Kinematic Viscosity Thermometer 207.5 to 212.5 F.

[a] Thermometers conforming to the requirements for A.S.T.M. Kinematic Viscosity Thermometers E 1 (28F–39T), E 1 (29F–39T), and E 1 (30F–39T) formerly prescribed in the Tentative Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1 – 42 T), 1942 Book of A.S.T.M. Standards, Part II, p. 1408, Part III, p. 1555, shall be considered as meeting these specifications.

[b] See Explanatory Note on Ice Point Determinations of Glass Thermometers which appears in the text.

TABLE I.—REQUIREMENTS FOR A.S.T.M. THERMOMETERS (*Continued*).

Name	A.S.T.M. Aniline Point[a]	A.S.T.M. Aniline Point[a]
A.S.T.M. Designation	E 1 (33C - 41T)	E 1 (34C - 41T)
Liquid	mercury	mercury
Filling above Liquid	nitrogen gas	nitrogen gas
Temperature Range	−38 to +42 C.	25 to 105 C.
Subdivisions	0.2 C.	0.2 C.
Total Length	403 to 409 mm. (15.87 to 16.10 in.)	403 to 409 mm. (15.87 to 16.10 in.)
Stem Diameter	6.0 to 7.0 mm. (0.236 to 0.276 in.)	6.0 to 7.0 mm. (0.236 to 0.276 in.)
Bulb Diameter	5.0 to 6.0 mm. (0.197 to 0.236 in.)	5.0 to 6.0 mm. (0.197 to 0.236 in.)
Bulb Length	10 to 20 mm. (0.39 to 0.79 in.)	10 to 20 mm. (0.39 to 0.79 in.)
Bottom of Bulb to Graduation Line at Distance	−38 C. 101 to 115 mm. (3.98 to 4.53 in.)	25 C. 101 to 115 mm. (3.98 to 4.53 in.)
Top of Thermometer to Graduation Line at Distance	42 C. 25 to 45 mm. (0.98 to 1.77 in.)	105 C. 25 to 45 mm. (0.98 to 1.77 in.)
Contraction Chamber	....	[b]
Expansion Chamber	yes	yes
Top Finish	glass ring	glass ring
Longer Graduation Lines at Each	1 C.	1 C.
Graduations Numbered at Each Multiple of	2 C.	2 C.
Immersion	51 mm. (2 in.)[c]	51 mm. (2 in.)[c]
Special Marking on Thermometer (See Section 7)	ASTM Aniline Point 51 mm. Imm.	ASTM Aniline Point 51 mm. Imm.
Scale Error at any point when standardized shall not exceed	0.2 C.	0.2 C.
Standardization	[d]	[d]
Marking on Case	A.S.T.M. Aniline Point Thermometer −38 to +42 C.	A.S.T.M. Aniline Point Thermometer 25 to 105 C.

[a] Editorially revised in 1942.

[b] The top of the contraction chamber shall be not more than 35 mm. (1.38 in.) above the bottom of the bulb. The mercury should stand at approximately the center of the contraction chamber at 0 C. (32 F.).

[c] A line around the stem 51 mm. (2 in.) above the bottom of the bulb shall be etched on the thermometer.

[d] The aniline point thermometers shall be standardized for 51-mm. or 2-in. immersion and for the following temperatures of the emergent mercury column:

−38 to +42 C.

Thermometer Reading	Average Temperature of Emergent Mercury Column
−35 C.	5 C.
−20 C.	15 C.
0 C.	20 C.
20 C.	25 C.
40 C.	30 C

25 to 105 C.

Thermometer Reading	Average Temperature of Emergent Mercury Column
25 C.	25 C.
50 C.	40 C.
75 C.	45 C.
100 C.	45 C.

TABLE I.—REQUIREMENTS FOR A.S.T.M. THERMOMETERS (*Continued*).

Name	A.S.T.M. Aniline Point[a]
A.S.T.M. Designation	E 1 (35C – 41 T)
Liquid	mercury
Filling above Liquid	nitrogen gas
Temperature Range	90 to 170 C.
Subdivisions	0.2 C.
Total Length	403 to 409 mm. (15.87 to 16.10 in.)
Stem Diameter	6.0 to 7.0 mm. (0.236 to 0.276 in.)
Bulb Diameter	5.0 to 6.0 mm. (0.197 to 0.236 in.)
Bulb Length	10 to 20 mm. (0.39 to 0.79 in.)
Bottom of Bulb to Graduation Line at Distance	90 C. 101 to 115 mm. (3.98 to 4.53 in.)
Top of Thermometer to Graduation Line at Distance	170 C. 25 to 45 mm. (0.98 to 1.77 in.)
Contraction Chamber	[b]
Expansion Chamber	yes
Top Finish	glass ring
Longer Graduation Lines at Each	1 C.
Graduations Numbered at Each Multiple of	2 C.
Immersion	51 mm. (2 in.)[c]
Special Marking on Thermometer (See Section 7)	ASTM Aniline Point 51 mm. I mm.
Scale Error at any point when standardized shall not exceed	0.2 C.
Standardization	[d]
Marking on Case	A.S.T.M. Aniline Point Thermometer, 90 to 170 C.

[a] Editorially revised in 1942.

[b] The top of the contraction chamber shall be not more than 35 mm. (1.38 in.) above the bottom of the bulb. The mercury should stand at approximately the center of the contraction chamber at 0 C. (32 F.).

[c] A line around the stem 51 mm. (2 in.) above the bottom of the bulb shall be etched on the thermometer.

[d] This thermometer shall be standardized for 51-mm. or 2-in. immersion and for the following temperatures of the emergent mercury column:

Thermometer Reading	Average Temperature of Emergent Mercury Column
100 C.	70 C.
130 C.	60 C.
160 C.	50 C.

TABLE I.—REQUIREMENTS FOR A.S.T.M. THERMOMETERS (*Continued*).

Name	A.S.T.M. Solvents Distillation	A.S.T.M. Solvents Distillation	A.S.T.M. Solvents Distillation
A.S.T.M. Designation	E 1 (37C - 44T)	E 1 (38C - 44T)	E 1 (39C - 44T)
Liquid	mercury	mercury	mercury
Filling above Liquid	nitrogen gas	nitrogen gas	nitrogen gas
Temperature Range	−2 to +52 C.	24 to 78 C.	48 to 102 C.
Subdivisions	0.2 C.	0.2 C.	0.2 C.
Total Length	380 to 385 mm. (14.96 to 15.16 in.)	380 to 385 mm. (14.96 to 15.16 in.)	380 to 385 mm. (14.96 to 15.16 in.)
Stem Diameter	6.0 to 7.0 mm. (0.236 to 0.276 in.)	6.0 to 7.0 mm. (0.236 to 0.276 in.)	6.0 to 7.0 mm. (0.236 to 0.276 in.)
Bulb Diameter	not greater than stem	not greater than stem	not greater than stem
Bulb Length	15 to 20 mm. (0.59 to 0.79 in.)	15 to 20 mm. (0.59 to 0.79 in.)	15 to 20 mm. (0.59 to 0.79 in.)
Bottom of Bulb to Graduation Line at Distance	−2 C. 125 to 145 mm. (4.92 to 5.71 in.)	24 C. 125 to 145 mm. (4.92 to 5.71 in.)	48 C. 125 to 145 mm. (4.92 to 5.71 in.)
Top of Thermometer to Graduation Line at Distance	52 C. 25 to 45 mm. (0.98 to 1.77 in.)	78 C. 25 to 45 mm. (0.98 to 1.77 in.)	102 C. 25 to 45 mm. (0.98 to 1.77 in.)
Contraction Chamber	none	[a]	[a]
Expansion Chamber	[b]	[b]	[b]
Top Finish	glass ring	glass ring	glass ring
Longer Graduation Lines at Each	1 C.	1 C.	1 C.
Graduations Numbered at Each Multiple of	2 C.	2 C.	2 C.
Immersion	100 mm. (3.94 in.)[c]	100 mm. (3.94 in.)[c]	100 mm. (3.94 in.)[c]
Special Marking on Thermometer (see Section 7)	ASTM Solvents Dist 100 mm. Imm.	ASTM Solvents Dist 100 mm. Imm.	ASTM Solvents Dist 100 mm. Imm.
Scale Error at any point when standardized shall not exceed	0.2 C.	0.2 C.	0.2 C.
Standardization	[d, e]	[d, e]	[d, e]
Marking on Case	A.S.T.M. Solvents Distillation Thermometer, −2 to +52 C., 100-mm. immersion	A.S.T.M. Solvents Distillation Thermometer, 24 to 78 C., 100-mm. immersion	A.S.T.M. Solvents Distillation Thermometer, 48 to 102 C., 100-mm. immersion

[a] The top of the contraction chamber shall be not more than 35 mm. (1.38 in.) above the bottom of the bulb. The mercury shall stand near the bottom of the contraction chamber at 0 C.

[b] The expansion chamber shall permit heating the thermometer at least 25 C. above the highest temperature on the scale.

[c] A line covering only one-eighth of the circumference of the stem shall be etched on the front of the thermometer at 100 mm. (3.94 in.) above the bottom of the bulb.

[d] For precautions to be observed in determining changes in bulb volume see Explanatory Note on Periodic Checking of Changes of Bulb Volume of Glass Thermometers, which appears in the text.

[e] The A.S.T.M. Solvents Distillation Thermometers shall be standardized for 100-mm (3.94-in.) immersion at the temperatures specified below and for the following average temperatures of the emergent mercury column:

Standardization Temperature	Average Temperature of Emergent Mercury Column	Standardization Temperature	Average Temperature of Emergent Mercury Column	Standardization Temperature	Average Temperature of Emergent Mercury Column
−2 to +52 C. Thermometer		24 to 78 C. Thermometer		48 to 102 C. Thermometer	
25 C	25 C.	25 C	25 C.	50 C	30 C.
50 C	25 C.	50 C	25 C.	75 C	30 C.
		75 C	25 C.	100 C	30 C.

TABLE 1.—REQUIREMENTS FOR A.S.T.M. THERMOMETERS (*Concluded*).

Name	A.S.T.M. Solvents Distillation	A.S.T.M. Solvents Distillation	A.S.T.M. Solvents Distillation
A.S.T.M. Designation	E 1 (40C - 44T)	E 1 (41C - 44T)	E 1 (42C - 44T)
Liquid	mercury	mercury	mercury
Filling above Liquid	nitrogen gas	nitrogen gas	nitrogen gas
Temperature Range	72 to 126 C.	98 to 152 C.	95 to 255 C.
Subdivisions	0.2 C.	0.2 C.	0.5 C.
Total Length	380 to 385 mm. (14.96 to 15.16 in.)	380 to 385 mm. (14.96 to 15.16 in.)	380 to 385 mm. (14.96 to 15.16 in.)
Stem Diameter	6.0 to 7.0 mm. (0.236 to 0.276 in.)	6.0 to 7.0 mm. (0.236 to 0.276 in.)	6.0 to 7.0 mm. (0.236 to 0.276 in.)
Bulb Diameter	not greater than stem	not greater than stem	not greater than stem
Bulb Length	15 to 20 mm. (0.59 to 0.79 in.)	15 to 20 mm. (0.59 to 0.79 in.)	15 to 20 mm. (0.59 to 0.79 in.)
Bottom of bulb to Graduation Line at Distance	72 C. 125 to 145 mm. (4.92 to 5.71 in.)	98 C. 125 to 145 mm. (4.92 to 5.71 in.)	95 C. 125 to 145 mm. (4.92 to 5.71 in.)
Top of Thermometer to Graduation Line at Distance	126 C. 25 to 45 mm. (0.98 to 1.77 in.)	152 C. 25 to 45 mm. (0.98 to 1.77 in.)	255 C. 25 to 45 mm. (0.98 to 1.77 in.)
Contraction Chamber	[a]	[a]	[a]
Expansion Chamber	[b]	[b]	[b]
Top Finish	glass ring	glass ring	glass ring
Longer Graduation Lines at Each	1 C.	1 C.	1 C.
Graduations Numbered at Each Multiple of	2 C.	2 C.	5 C.
Immersion	100 mm. (3.94 in.)[c]	100 mm. (3.94 in.)[c]	100 mm. (3.94 in.)[c]
Special Marking on Thermometer (See Section 7)	ASTM Solvents Dist 100 mm. Imm.	ASTM Solvents Dist 100 mm. Imm.	ASTM Solvents Dist 100 mm. Imm.
Scale Error at any point when standardized shall not exceed	0.2 C.	0.2 C.	0.5 C.
Standardization	[d], [e]	[d], [e]	[d], [e]
Marking on Case	A.S.T.M. Solvents Distillation Thermometer, 72 to 126 C., 100-mm. immersion	A.S.T.M. Solvents Distillation Thermometer, 98 to 152 C., 100-mm. immersion	A.S.T.M. Solvents Distillation Thermometer, 95 to 255 C., 100-mm. immersion

[a] The top of the contraction chamber shall be not more than 35 mm. (1.38 in.) above the bottom of the bulb. The mercury shall stand near the bottom of the contraction chamber at 0 C.

[b] The expansion chamber shall permit heating the thermometer at least 25 C. above the highest temperature on the scale.

[c] A line covering only one-eighth of the circumference of the stem shall be etched on the front of the thermometer at 100 mm. (3.94 in.) above the bottom of the bulb.

[d] For precautions to be observed in determining changes in bulb volume see Explanatory Note on Periodic Checking of Changes of Bulb Volume of Glass Thermometers, which appears in the text.

[e] The A.S.T.M. Solvents Distillation Thermometers shall be standardized for 100-mm. (3.94-in.) immersion at the temperatures specified below and for the following average temperatures of the emergent mercury column:

Standardization Temperature	Average Temperature of Emergent Mercury Column
72 to 126 C. Thermometer	
75 C.	30 C.
100 C.	30 C.
125 C.	30 C.
98 to 152 C. Thermometer	
100 C.	30 C.
125 C.	35 C.
150 C.	35 C.
95 to 255 C. Thermometer	
100 C.	30 C.
150 C.	35 C.
200 C.	40 C.
250 C.	45 C.

Tentative Method of Test for

PARTICLE SIZE DISTRIBUTION OF SUBSIEVE SIZE PARTICULATE SUBSTANCES[1]

A.S.T.M. Designation: E 20 - 33 T

ISSUED, 1933.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. This method of test covers the procedure for determining the particle size of particulate substances in absolute units, as far as the particle shape will permit. It is intended to cover the range of sizes between the 74-micron (No. 200) sieve and 0.2 micron. The method is applicable in its entirety to homogeneous materials. In the case of mixtures, the extent of application is limited by the properties of the components of the mixture.

Definitions

2. (*a*) *Dispersion.*—In microscopy it refers to the distribution of the individual particles. In statistical work it is measured by the standard deviation.

NOTE.—Both uses of the word seem too generally accepted to warrant a change in either.

(*b*) *Individual Particle.*—Those minute units of matter (of which the material is composed) whose size and shape depend only on the force of cohesion. Such particles cannot be subdivided without separating like molecules that are within the range of the cohesive attraction of one another.[3]

(*c*) *Aggregate.*—A group of two or more attached particles.

NOTE.—There are three forms of aggregates: namely, ultimate working unit, flocculate, and apparent flocculate.

(*d*) *Ultimate Working Unit.*—An individual particle or any group of individual particles that are so firmly held together by forces of adhesion that they remain intact as a group throughout the duration of their application.

(*e*) *Flocculate.*—Flocculation occurs only where particles have been incorporated in a liquid or plastic medium. A flocculate is a group of particles held together by a force apparently arising from interfacial tension. This force of flocculation is relatively weak, consequently flocculates do not function as large individual particles, and cannot be classified under ultimate working units.

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee E-1 on Methods of Testing.

[2] Accepted by the Society at annual meeting, June, 1933. Editorially revised and rearranged in 1939.

[3] Henry Green, *Journal of Industrial Hygiene*, Vol. 7, p. 155 (1925).

Under slight, continuously applied force flocculates are constantly breaking down and reforming.

Apparent Flocculates as found in gas-floated or air-floated particles show a somewhat similar type of aggregation. Usually the force of attraction between the particles is relatively weak and the particles can readily be dispersed in suitable media. For practical purposes, the more inclusive term, aggregate, is preferable to flocculate for gas-floated or air-floated particles.

(*f*) *Average Diameter.*—The diameter of a hypothetical particle which, in some particular way, represents the total mass of particles.

(*g*) *Nonuniformity.*—A nonuniform particulate substance is one in which the constituent particles differ from one another in diameters.

Rough Separation of Sample into Size Groups

3. (*a*) For pigments or other materials which are essentially less than 6 microns, separation of the sample is not necessary and the mount shall be prepared in accordance with Section 4.

(*b*) For wide ranges of distribution of particle size in subsieve size portions, auxiliary separation into groups of sizes is necessary to facilitate measurement with the microscope. Rough separation is permissible as exact size measurement in microns follows. The following general ranges of particle diameter are proposed for the separation:

A.....	24 microns and larger
B.....	24 to 12 microns
C.....	12 to 6 microns
D.....	6 to 0.2 microns

For specific purposes A or D may be further separated.

NOTE.—If the sample contains particles larger than the 74-micron (No. 200) sieve, the material shall be separated by sieving wet in a suitable fluid. The portion which passes the sieve may be recovered by evaporation of the fluid. The portion which remains on the sieve may be subjected when dry to a standard sieve test and additional material that passes the 74-micron (No. 200) sieve added to that which is to be tested.

(*c*) Separation of the sample shall be made by air or liquid elutriation or by sedimentation at room temperature. The volume of liquid in the elutriator or sedimentation apparatus should be between 20 and 100 ml. per gram of solid. With finer particles and with material of lesser density the greater volumes should be used. The technique involved in the elutriation or settling is necessarily influenced by the type of material being measured and the dispersing liquids. The size of the vessel and dispersion will be specified in terms of the uniformity obtained. The microscope measurement will be used as a check upon the effectiveness of the separation. For accurate results at least 50 per cent by weight must fall within the boundaries of the suggested ranges set for the elutriation. Of the material falling outside the boundaries, not over 5 per cent by weight may fall outside of a range which is one half of the lower limit and twice the upper limit of the range under consideration.

(*d*) The fluid used in the elutriation or sedimentation shall contain no dissolved solids which would influence the weight determination of a given portion, that is, the fluid shall be 100 per cent volatile. It shall not react chemically with the material being elutriated. Water, kerosine, alcohols, and the like may be used. It is suggested that the filtering of any elutriator portion should be through alundum to avoid contamination of the sample with paper or pulp fibers. As an alternative, the solid may be recovered from each stage of the elutriator by evaporation of a concentrated slurry,

provided the temperatures used for the evaporation do not alter the specific product. The total weight of the portions recovered shall check within 5 per cent the weight of material initially placed in the elutriator.

(*e*) All limits are specified to insure good microscopic technique. Good elutriation technique for analytical purposes falls well within these limits.

NOTE.—Quite obviously, materials containing components of different densities will elutriate or settle in ranges of low uniformity. The present requirements can only be applied when the material meets the requirements for homogeneity. If the materials can be readily identified under the microscope, they may be measured individually and the results considered as exact, notwithstanding wide differences in density between two materials which may cause lack of uniformity. In other cases, such as cement or coal, where the presence respectively of gypsum and ash constituents may affect the test, these effects may be considered negligible for the normal type of products.

Preparation of Mount

4. Any one of a number of methods of preparing the mount may be used, the criterion of suitability being conformity to the following requirements:

The particles shall be essentially in one plane.

The particles shall be free from motion.

The particles shall be dispersed, showing individual grains instead of aggregates and flocculates.

The particles shall not be ground in mounting.

The mount shall be truly representative of the distribution of sizes in the material.

The mounting medium shall be selected to give maximum definition.

(*a*) *Fine Materials, Pigments, etc.*—The type of mount that should be used depends on the physical properties of the particles, and for this reason no definite requirements are specified. The general procedure shall be to place the material being tested on a microscope slide, and then to rub it out in a solution of gum or resin in a solvent until the particles are well dispersed. After the solvent has evaporated, the nonvolatile constituent of the dispersing agent serves to cement the particles to the slide in the dispersed state.

NOTE.—Detailed technique will be found in the work of Dunn[4] and Green.[5] The former uses balsam in xylol, and the latter turpentine and dammar. Other mounting media suggested are 0.5 per cent glycerol in alcohol, rubber cement, styrax in xylol, and the chlorinated naphthalenes and saponin.

(*b*) *Coarse Material, Ground Products, etc.*—In the case of coarse materials it is often advantageous to use the so-called dry mount, in which the dispersing agent used is volatile and can be driven off after the material has been dispersed on the slide. The uniformity of the mount when examined microscopically is the best criterion of an acceptable mount.

Procedure

5. (*a*) *Direct Observation Method.*—Direct observations using the Filar micrometer[6] has been used with fair success and reasonable degree of accuracy on the coarser fraction of particulate substances. The Filar micrometer is an attachment which fits into the draw tube of the microscope and is fitted with a Ramsden-type eyepiece which permits focusing on a movable cross hair activated by a micrometer screw. This intrument permits direct measurement of the particle as viewed on the slide. This method, however, is

[4] E. J. Dunn, Jr., *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 2, p. 15 (1930).
[5] Henry Green, *Industrial and Engineering Chemistry*, Vol. 16, No. 7, p. 667 (1924).
[6] J. C. Pearson and W. H. Sligh, National Bureau of Standards *Technological Paper No. 48* (1915).

not recommended for materials in the 0.2 to 6 micron class size.

(*b*) *Projection Method.*—The projection method[4] which involves throwing the image of the particle on a screen has several distinct advantages. It further magnifies the image and permits focusing through the depth of the mount, insuring a proper focus and count of all the particles.

(*c*) *Photomicrographic Method.*—The photomicrographic method,[7,8] which involves taking a photomicrograph of the particles and subsequently measuring them on a print or by projecting the negative on the screen, is also recommended. This method has the very decided advantage of making a permanent record of the sample and permits eye comparisons of two or more samples. In the use of this method it is extremely important that the mount be made according to the procedure outlined under the method of Green[7] and fulfill the requirements specified there.

Measurement of Diameter

6. In each of the three procedures referred to in Section 5 the horizontal diameter shall be measured unless the particles are very uniform in shape, and in that event a diameter which most nearly represents the average shall be selected. For irregularly shaped particles, the horizontal diameter splitting the particle in half in one direction consistently maintained has been shown[9] to give fairly consistent and accurate results. For flat plates, it is necessary to consider the third dimension for accurate results. In any case, in reporting results, the diameter measured shall be specified.

[7] Henry Green, *Journal*, Franklin Inst., Vol. 192, p. 637 (1921).
[8] Sheppard and Lambert—not yet published.
[9] Martin, *Journal*, Am. Ceramic Soc., Vol. 23, p. 6 (1924).

Scale Limits for Measurement

7. (*a*) If more than 10 per cent by weight is coarser than 6.00 microns, the sample shall be elutriated prior to microscopic measurement. A graded scale for microscopic sizing is as follows:

Diameter Range in Microns	
General Case (20 per cent or more under 6 microns)	Special Case (Less than 20 per cent under 6 microns)
72 to 60	
60 to 48	
48 to 36	
36 to 24	
24 to 18	
18 to 12	
12 to 9	
9 to 6	
	Same Scale and
6.00 to 5.00	6 to 4.5
5.00 to 4.00	4.5 to 3
4.0 to 3.5	3 to 1.5
3.5 to 3.0	1.5 to 0
3.0 to 2.5	
2.5 to 2.0	
2.0 to 1.8	
1.8 to 1.6	
1.6 to 1.4	
1.4 to 1.2	
1.2 to 1.0	
1.0 to 0.9	
0.9 to 0.8	
0.8 to 0.7	
0.7 to 0.6	
0.6 to 0.5	
0.5 to 0.4	
0.4 to 0.3	
0.3 to 0.2	
0.2	

(*b*) Two hundred and fifty particles shall be measured in each of three fields taken at random.

(*c*) The magnification shall be dependent on the class size being measured, and shall be sufficient to permit the differentiation specified above.

Expression of Results

8. Results shall be expressed using one or more of the following average diameters:

Average Diameter	To Be Used:	Formula
$\bar{d}_1$	As arithmetical mean.....	$\bar{d}_1 = \frac{\Sigma nd}{\Sigma n}$
$\bar{d}_3$	To determine S_p when p is known	$\bar{d}_3 = \frac{\Sigma nd^3}{\Sigma nd^2}$

Average Diameter	To Be Used:	Formula
$\bar{d}_4$	To determine volume or weight distribution....	$\bar{d}_4 = \frac{\Sigma nd^4}{\Sigma nd^3}$
$\bar{D}$	To determine N when ρ is known	$\bar{D} = \sqrt[3]{\frac{\Sigma nd^3}{\Sigma n}}$
Δ	To determine S when N is known	$\Delta = \sqrt{\frac{\Sigma nd^2}{\Sigma n}}$
M_g	As geometric mean or median.....	$\log M_g = \frac{\Sigma n \log d}{\Sigma n}$

SYMBOLS:

S_m = specific surface in square meters per gram,
S_v = specific surface in square meters per cubic centimeter,
N_m = number of particles per gram,
N_v = number of particles per cubic centimeter,
Σn = number of particles in any given mass,
ρ = density of the material,
θ = arithmetic standard deviation,
θ_g = geometric standard deviation, and
P.E. = probable error.

Report

9. (*a*) If 90 per cent by weight fall in any one class size, a frequency curve shall be given.

(*b*) In the 0 to 6-micron class, either a frequency curve or the distribution by weight shall be reported.

(*c*) In elutriated products, weight distribution shall be expressed and reported as follows: Weight distribution shall be the basis of comparison for all elutriated samples. The weight distribution for each elutriated portion shall be calculated on the basis of spherical particles, using the arithmetic mean of the microscopic class interval as the average diameter for all particles in that range. Each of the elutriated fractions shall total in weight distribution to the percentage retained in the elutriator and the composite curve shall be a summation of the distribution values in each range. The degree of overlapping is a measure of the efficiency of the elutriation, and it shall not be abnormal in amount.

Tentative Method of Test for

SOFTENING POINT[1]

BALL AND SHOULDERED RING APPARATUS

A.S.T.M. Designation: E 28 - 42 T

ISSUED, 1936; REVISED, 1939, 1942.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. (*a*) This method of test for softening point by ring-and-ball method is applicable to asphalts, tars, pitches, rosins, and most resins both natural and synthetic.

(*b*) *Softening Point.*—In general, with materials of this type, softening does not take place at a definite temperature. As the temperature rises, these materials gradually and imperceptibly change from brittle or exceedingly thick and slow flowing materials to softer and less viscous liquids. For this reason the determination of the softening point must be made by a fixed, arbitrary, and closely defined method if the results obtained are to be comparable.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Ring.*—A brass shouldered ring conforming to the dimensions (Note) shown in Fig. 1 (*a*). The ring may be attached, by brazing, to a No. 11 B. & S. gage brass wire (diameter 2.31 mm. = 0.091 in.), (see Fig. 2).

NOTE.—The tapered ring formerly specified in the Tentative Method of Test for Softening Point (Ball and Tapered Ring Apparatus) (A.S.T.M. Designation: E 28 - 39 T) of the American Society for Testing Materials,[3] may be used interchangeably with the shouldered ring.

(*b*) *Ball.*—A steel ball, 9.53 mm. ($\frac{3}{8}$ in.) in diameter weighing between 3.45 and 3.55 g.

(*c*) *Ball Guide.*—A ball guide for centering the balls, constructed of brass, and having the shape and dimensions illustrated in Fig. 1 (*c*).

(*d*) *Container.*—A glass vessel, capable of being heated, not less than 8.5 cm. ($3\frac{1}{3}$ in.) in diameter and not less than 10.5 cm. ($4\frac{1}{8}$ in.) in depth from the bottom of the flare. (A 600-ml. low-form Griffin beaker meets this require-

[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee E-1 on Methods of Testing.

[2] The ring-and-ball method was first standardized by the Society's Committee D-4 on Road and Paving Materials as applying to bituminous materials and issued under the A.S.T.M. Designation D 36. The present method is an extension and modification of the ring-and-ball method to make it applicable to materials other than bituminous materials as well.

[3] 1939 Book of A.S.T.M. Standards, Part II, p. 1169; Part III, p. 1127.

ment. For use with the ring holder an 800-ml. tall-form beaker is convenient.)

(*e*) *Support for Ring and Thermometer.*—Any convenient method for supporting the ring and thermometer may be used (see Figs. 2, 3, and 4) provided it meets the following requirements: The ring shall be supported in a substantially horizontal position. The top of the ring shall be at least 7.6 cm. (3.0 in.) below the top of the container and at least 5.1 cm. (2.0 in.) below the surface of the heating liquid. Using the apparatus in Fig. 1, the bottom of the ring shall be 2.5 cm. (1.0 in.) above the bottom of the glass vessel. If the ring holder is used (see Fig. 3), the bottom of the ring shall be 2.5 cm. (1.0 in.) above the upper surface of the lower horizontal plate; the lower horizontal plate shall be at least 1.3 cm. (½ in.) above the bottom of the glass vessel. The thermometer shall be suspended so that the bottom of the bulb is level with the bottom of the ring and within 1.0 cm. (⅜ in.) but not touching the ring. For

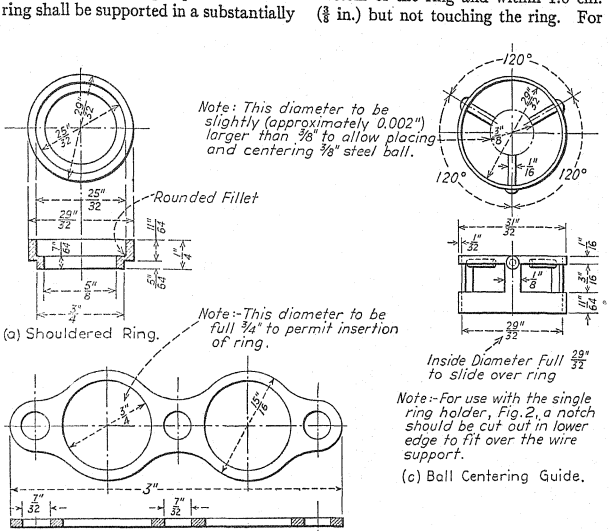


FIG. 1.—Shouldered Ring, Ring Holder, and Ball-Centering Guide.

referee work the ring holder shall not carry more than two rings.

(*f*) *Thermometers.*—(*1*) An A.S.T.M. Low Softening Point Thermometer, graduated in either Centigrade or Fahrenheit degrees as specified, having a range of −2 to +80 C. or +30 to +180 F. and conforming to the requirements for this thermometer as prescribed in the Standard Specifications for A.S.T.M.

Thermometers (A.S.T.M. Designation: E 1) of the American Society for Testing Materials.[4]

(*2*) An A.S.T.M. High Softening Point Thermometer, graduated in either Centigrade or Fahrenheit degrees as specified, having a range of 30 to 200 C. or 85 to 392 F. and conforming to the requirements for this thermometer as prescribed in Standard Specifications E 1.[4]

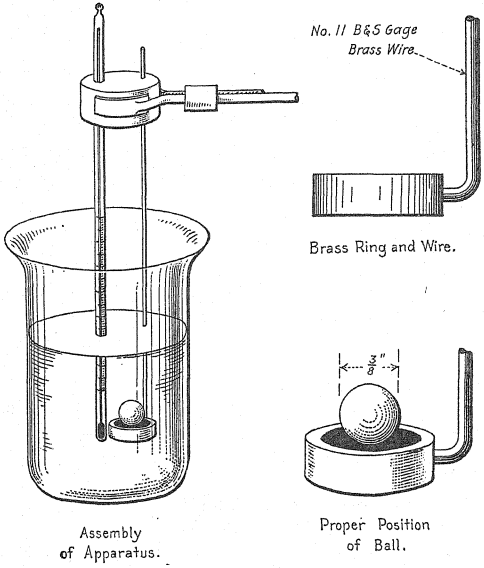


FIG. 2.—Assembly of Apparatus, Showing Single Shouldered Ring.

Preparation of Sample

3. (*a*) The sample shall be selected from the material to be tested in such a manner as will avoid the inclusion of the surface layer. A quantity at least twice that necessary to fill the desired number of rings, and in no case less than 40 g., shall be melted immediately in a clean container, using an oven, hot plate, sand or oil bath to prevent local overheating. Care shall be taken to avoid incorporating air bubbles in the sample which shall not be heated above the temperature necessary to pour the material readily without inclusion of air bubbles. The time from the beginning of heating to the pouring of the sample shall not exceed 15 min.

(*b*) Immediately before filling the rings, they shall be preheated to ap-

[4] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

proximately the temperature at which the material is to be poured. The rings, while being filled, should rest on an amalgamated brass plate. The quantity of material poured into the rings shall be such that after 20-min. cooling at room temperature of materials with softening points below 80 C. (176 F.) and 40-min. cooling for materials of higher softening point, an excess amount will remain which shall then be cut off cleanly with a slightly heated spatula. For routine testing this period of time may be curtailed according to the characteristics of the material

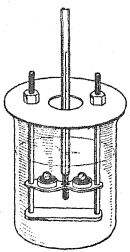


FIG. 3.—Assembly of Apparatus, Showing Two Rings.

under test. In case the test is to be repeated, a clean container and fresh sample shall be used.

Procedure

4. (*a*) *Assembling Apparatus.*—The glass container shall be filled to a depth not less than 9 cm. (3.5 in.) with freshly boiled distilled water (or with glycerol for materials melting above 80 C. (176 F.)) which has been cooled to not less than 45 C. (113 F.) below the anticipated softening point but in no case lower than 5 C. (41 F.) for materials melting below 80 C. (176 F.) and in no case lower than 35 C. (95 F.) for materials melting above 80 C. (176 F.). Provision shall be made for centering the ball on the upper surface of the sample, either by use of the ball guide or by making a slight indentation in the center of the sample. The latter may be done by pressing the ball or a rounded rod into the material at this point. In the case of hard materials the rod may be heated. The ring containing the sample and ball guide, if used, shall be suspended in the water so that the lower surface of the

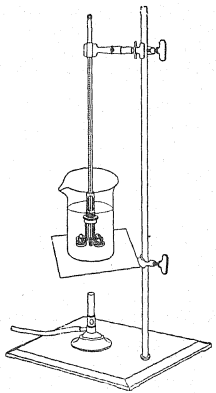


FIG. 4.—Assembly of Apparatus, Showing Four Rings.

filled ring is 2.5 cm. (1 in.) above the bottom of the glass container (Fig. 2) or 2.5 cm. above the upper surface of the lower horizontal plate which is at least 1.3 cm. ($\frac{1}{2}$ in.) above the bottom of the glass vessel (Fig. 3) and so that its upper surface is at least 5.1 cm. (2 in.) below the surface of the water. The ball shall be placed in the water but not on the specimen. The thermometer shall be suspended so that the bottom of the bulb is level with the bottom of the ring and within 1.0 cm. ($\frac{3}{8}$ in.) but not touch-

ing the ring. The initial temperature shall be maintained for 15 min. With suitable forceps the ball shall be placed in the center of the upper surface of the material in the ring.

(*b*) *Heating.*—To facilitate uniform heating, the burner shall be placed midway between the center and the edge of the beaker on a diameter at right angles to the diameter which includes the ring or rings and the thermometer bulb. The effect of drafts on uniformity of heating must be eliminated, using shields if necessary. The heat shall be applied in such manner that the temperature of the bath is raised 5 C. (9 F.) each minute.

(*c*) *Permissible Variation in Rise of Temperature.*—The rate of rise of temperature shall be uniform and shall not be averaged over the period of the test. The maximum permissible variation for any minute period after the first three shall be plus or minus 0.5 C. (0.9 F.). All tests in which the rate of rise exceeds these limits shall be rejected.

Softening Point

5. (*a*) The temperature reading to one-half of the smallest scale division indicated by the thermometer at the instant the sample touches the bottom of the container (or the lower horizontal plate) shall be taken as the softening point. No corrections shall be made for emergent stem of the thermometer.

(*b*) For materials softening around 80 C. (176 F.) the nature of the bath fluid (water or glycerol) shall be reported, since a glycerol bath yields slightly higher results than a water bath.

Precautions

6. (*a*) The use of freshly boiled distilled water is essential, as otherwise air bubbles may form on the specimen and affect the results. Rigid adherence to the prescribed rate of heating is absolutely essential for reproducibility of results.

(*b*) A thin, amalgamated copper plate or a sheet of filter paper placed on the bottom of the glass vessel will prevent the material from sticking to the bottom of the glass vessel, thereby saving considerable time and trouble in cleaning.

(*c*) In order to insure uniform heat distribution at all times throughout the bath, a mechanical stirrer should be used.

Reproducibility of Results

7. With care and proper attention to details, duplicate determinations of softening point by this method should not differ by more than 1.0 C. (1.8 F.).

Tentative Recommended Practices for

DESIGNATION OF NUMERICAL REQUIREMENTS IN STANDARDS[1]

A.S.T.M. Designation: E 29 – 40 T

ISSUED, 1940.[2]

These Tentative Recommended Practices of the American Society for Testing Materials are, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. (*a*) These recommended practices are intended to assist the various standing committees in the use of uniform terms and conventions in expressing numerical requirements in specifications and methods of testing. Their aim is to outline practices which should aid in clarifying the intended meaning of limiting values, with which test values are compared in acceptance and rejection of material. Certain nonnumerical terms closely related to numerical expressions are also included.

(*b*) These practices are not intended to be used in the interpretation of specifications which do not definitely call for their use. The provisions of these practices apply to the Society's standards as they bear on the acceptance and rejection of material, however, when such standards make definite reference to the practices as covered in Section 2.

(*c*) Definite interpretations are assigned to the following:

(*1*) The phrase "to the nearest ....." when applied to a specified limit (see Section 4 (*a*)).

(*2*) The underlining of figures in a specified limit, for example, "1.500" (see Section 5 (*a*)).

(*3*) The symbol "±" when applied to a specified value of maximum permissible deviation (see Section 8 (*b*)).

(*4*) The term "average" (see Section 9 (*a*)).

Reference to These Practices in Standards

2. The inclusion of the following paragraph, or a suitable equivalent, in any standard (preferably immediately after the section on "Scope") shall constitute due notification that numerical requirements in that standard are subject to the interpretations set forth in these recommended practices:

Interpretation of Numerical Requirements.—The numerical requirements of this standard are subject to the interpretations set forth in the

[1] Under the standardization procedure of the Society, these recommended practices are under the jurisdiction of the A.S.T.M. Committee E-1 on Methods of Testing.

[2] Accepted by the Society at annual meeting, June, 1940.

Tentative Recommended Practices for Designation of Numerical Requirements in Standards (A.S.T.M. Designation: E 29) of the American Society for Testing Materials. Particular attention is called to the use of—(insert one of the following: (*1*) the phrase "to the nearest ....."; (*2*) underlining; (*3*) underlining and use of the phrase "to the nearest .....")—in expressing specified limits.

Expression of Numerical Requirements

3. (*a*) The unqualified statement of a numerical limit, such as "2.50 in. max.", cannot, in view of different established practices and customs, be regarded as carrying a definite operational meaning (*1*) as to the number of places of figures to be read on the scale of measurement, or (*2*) as to the number of places of figures to be retained in an observed or calculated value for purposes of acceptance and rejection.

Measurement.—In some fields, specified values of 2.5 in. max., 2.50 in. max., 2.500 in. max., are taken to imply that a length determination should be made to the nearest 0.1 in., 0.01 in., 0.001 in., respectively. In other fields, no such significance is attached to this difference of expression.

Acceptance and Rejection.—In some fields, specified values of 2.5 in. max., 2.50 in. max., 2.500 in. max., are taken to imply that for purposes of acceptance and rejection, observed values or calculated values should be rounded off to the nearest 0.1 in., 0.01 in., 0.001 in., respectively. In other fields, these three specified values are taken to imply the same absolute limit of two and one-half inches and that no deviation, however small, in excess of this value is allowed.

(*b*) Clear meaning on these points can be assured only by specifying the following operations:

(*A*) *How measurement values are to be obtained, and*

(*B*) *How the observed values or calculated values are to be handled when they are compared to specified values in determining acceptability of material.*

(*c*) In specifying operation (*A*) the following items should be included:

(*1*) The test or analytical method to be followed,

(*2*) The type of measuring device to be used,

(*3*) The units of measurement to be used, and

(*4*) The number of places of figures to be read on the scale of measurement.

(*d*) Methods of specifying operation (*B*) are given in the following Paragraphs (*e*) to (*g*).

(*e*) The numerical requirements should be so expressed that the number of places of figures to be considered significant for purposes of acceptance and rejection are clearly indicated.

(*f*) If *all* places of figures in an observed or calculated value are to be considered significant for purposes of acceptance and rejection, this intent may be made clear by a direct statement in the standard, such as: "Observed or calculated values should not be rounded off but should be compared directly with specified values, for purposes of acceptance and rejection."

(*g*) If a *limited* number of places of figures in an observed or calculated value are to be considered significant for purposes of acceptance and rejection, this intent may be expressed by the use of the phrase "to the nearest ....." as follows:

Carbon (to the nearest 0.01 per cent)..... 0.20 per cent, min.
Tensile strength (to the nearest 100 psi.)..... 62 000 psi., min.

When the use of the phrase "to the nearest....." is not practicable, the number of figures to be considered significant may be indicated by a direct statement or by underlining the significant places, as follows:

Carbon..... 0.20 per cent, min.
Tensile strength..... 62 000 psi., min.

Meaning of Phrase "to the nearest....."

4. (*a*) The phrase, "to the nearest 10, 1, 0.1, 0.01, etc.," when applied to specified limits, shall be interpreted to have the meaning that an observed value or a calculated value is, for purposes of acceptance and rejection, to be rounded off to the nearest unit in the place occupied by the digit 1 in the phrase "to the nearest.....";" that is, shall be rounded off to the nearest digit in the last right-hand place of figures considered significant.

Specified Limit		Observed Value or Calculated Value	Rounded-off Value to be Used for Acceptance Purposes	Conforming to Specified Limit
Tensile Strength (to the nearest 100 psi.)	60 000 psi., min.	59 940	59 900	No
		59 950	60 000	Yes
		59 960	60 000	Yes
Tensile Strength (to the nearest 100 psi.)	60 500 psi., min.	60 440	60 400	No
		60 450	60 400	No
		60 460	60 500	Yes
Antimony (to the nearest 0.01 per cent)	0.90 per cent, min.	0.894	0.89	No
		0.895	0.90	Yes
		0.896	0.90	Yes
Antimony (to the nearest 0.01 per cent)	0.91 per cent, min.	0.904	0.90	No
		0.905	0.90	No
		0.906	0.91	Yes

NOTE.—The final rounded-off value should be obtained from the most precise value available and not from a series of successive rounded-off values; for example, 59,490 psi. to the nearest 100 and then to the nearest 1000 psi. becomes 59,500 and 59,000 (not 60,000) psi., respectively, since the most precise value available is less than 59,500.

(*b*) The actual rounding-off procedure shall be carried out as follows:[3]

(*1*) When the figure next beyond the last figure or place to be retained is less than 5, the figure in the last place retained should be kept unchanged.

(*2*) When the figure next beyond the last figure or place to be retained is more than 5, the figure in the last place retained should be increased by 1.

(*3*) When the figure next beyond the last figure or place to be retained is 5, and

(*a*) there are no figures, or only zeros, beyond this 5, if the figure in the last place to be retained is odd, it should be increased by 1; if even, it should be kept unchanged;

(*b*) if the 5 next beyond the figure in the last place to be retained is followed by any figures other than zero, the figure in the last place retained should be increased by 1, whether odd or even.

The above rules for rounding off may be restated as follows:

(*A*) The figure in the last place to be retained should be kept unchanged (*a*) when the figure in the next place is less than 5; (*b*) when the figure in the next place is 5 followed by no other figures or by only zeros, and the figure in the last place retained is even.

(*B*) The figure in the last place to be retained should be increased by 1, (*a*) when the figure in the next place is more than 5; (*b*) when the figure in the next place is 5 followed by no other figures or by only zeros, and the figure in the last place retained is odd; (*c*) when the figure in the next place is 5, followed by any figure or figures other than zero.

(*c*) If it should be necessary to round off to the nearest 50, 5, 0.5, 0.05, etc., this can be done by the application of the same general principles used above except that in case of observed or calculated values that are exactly half-way between two rounded-off values, the rounding off is done step-wise, that is, in two successive steps; for example, 6025 to the nearest 50 first becomes 6020 (Section 4 (*b*), item (3) (*a*)), which in turn becomes 6000; while 6075 to the nearest 50 first becomes 6080 (Section 4 (*b*), item (3) (*a*)), which in turn becomes 6100.

Meaning of the "Underlining" Symbol

5. (*a*) Underlining, when applied to specified limits shall be interpreted to

[3] This rounding-off procedure agrees with that adopted in the American Standard Rules for Rounding Off Numerical Values (ASA No: Z25.1—1940).

have the meaning that an observed value or a calculated value is, for purposes of acceptance and rejection, to be rounded off to the nearest unit in the last right-hand place of figures underlined.

(*b*) Thus underlining is the simple equivalent of the phrase "to the nearest 10, 1, 0.1, 0.01, etc."

Use of the Phrase "to the nearest...." and of the Underlining Symbol

6. (*a*) The use of the phrase "to the nearest....." (or of underlining) in designating specified limits, in effect specifies that the operation of "rounding off" observed or calculated values shall be considered applicable in determining whether those values are conforming or nonconforming with the specified limit.

(*b*) It is recommended that the rounding-off method as indicated by the use of the phrase "to the nearest....." (or by underlining) be employed as widely as possible in designating limits for dimensions. In view of the fact that well established gage tolerances and other practices in some fields do not employ the principle of "rounding off" here discussed, the use of the phrase "to the nearest....." (or of underlining) may not be considered universally applicable to all dimensional requirements. Where other procedures are to be used, it is recommended that the standard contain a direct statement as to the practice intended by the committee originating the specification. Clear expression on this point should aid in preventing misunderstandings and disagreements in instances where observed values are marginally close to specified values.

(*c*) It is recommended that the phrase "to the nearest....." (or underlining) be used generally in designating percentage chemical composition requirements, and that the number of decimal places used in expressing limiting values be the same as the number of decimal places to be considered significant for purposes of acceptance and rejection.

(*d*) In the interests of simplicity of expression, a general statement, used as a caption or a footnote, should be used in preference to repetitions of the phrase "to the nearest....." (or to repeated underlining) in instances where the "rounding-off" method is to be specified for a large number of similar requirements. Suggestions as to how this may be accomplished are given in the following examples:

(*1*) If the rounding-off method is to apply to all specified limits in the standard and *if all expressed figures are to be considered significant*, this may be indicated by adding the following sentence to the notification paragraph given in Section 2, as follows:

The following applies to all specified limits in this standard: For purposes of acceptance and rejection, an observed value or a calculated value should be rounded off "to the nearest unit" in the last right-hand place of figures used in expressing the limiting value.

(*2*) If the rounding-off method is to apply to all specified limits in a table, this may be indicated by a caption or by a footnote in the manner indicated in the following examples:

Example 1.—Same significant places for all items in a table (illustrating the use of a caption).

	Chemical Composition per cent (to the nearest 0.1 per cent)
Copper	4.5 ± 0.5
Iron	1.0 max.
Silicon	2.5 ± 0.5
Other constituents (magnesium + zinc + manganese)	0.5 max.
Aluminum	remainder

Example 2.—Same significant places for all items in a table, with one or two exceptions (illustrating the use of a caption and a footnote).

	Chemical Composition. per cent (to the nearest 0.1 per cent)
Aluminum	6.0 ± 0.7
Zinc	3.0 ± 0.5
Manganese	0.15 min.[a]
Copper	0.05 max.[a]
Silicon	0.5 max.
Other constituents (cadmium + tin + nickel) (nickel 0.03 max.[a])	0.3 max.
Magnesium	remainder

[a] To the nearest 0.01 per cent.

Example 3.—Significant places not the same for all items in a table (illustrating the use of a footnote).

	Chemical Composition, per cent	
	Min.	Max.
Nickel	57	....
Chromium	14	18
Manganese	....	3
Silicon	....	0.40
Carbon	....	0.25
Sulfur	....	0.03
Iron	remainder	

NOTE.—The following applies to all numerical requirements in this table: For purposes of acceptance and rejection, an observed value or a calculated value obtained from an analysis should be rounded off "to the nearest unit" in the last right-hand place of figures used in expressing the limiting value.

Decimals

7. Decimals are preferred to fractions in expressing numerical requirements, where practicable. It is recognized that the use of inches and fractions of inches is standard practice in specifying dimensions for some materials and products.

Limits

8. (*a*) Specifications should indicate limits to the range of permissible variations of acceptable material. These may be expressed by the use of maximum and minimum values, or permissible plus and minus deviations from a specified value.

(*b*) *Double Limits With Preference.*—If there is a preferred value, it should be indicated together with maximum and minimum values, or permissible plus and minus deviations.

Example:

Antimony (to the nearest 0.01 per cent) ..... { 0.98 per cent, preferred } { 0.90 per cent, min. / 1.10 per cent, max. }

or

Antimony (to the nearest 0.01 per cent) ..... { 0.98 per cent, preferred } { −0.08 per cent / +0.12 per cent }

If the preferred value lies half-way between limits, the maximum deviations permissible may be expressed by the phrase "plus or minus" or the "±" sign. An expression using the phrase "plus or minus" or the sign "±" shall be interpreted to indicate a preferred value, and is thus to be considered as having a different meaning from the statement of merely a maximum and a minimum value (see Paragraph (*c*)).

Example:

Antimony (to the nearest 0.01 per cent).... { 1.00 ± 0.10 per cent }

(*c*) *Double Limits Without Preference.*—If there is no preference for a specific value, maximum and minimum values alone should be indicated.

Example:

Weight (to the nearest 0.01 lb.)..... { 23.40 lb., min. / 23.50 lb., max. }

(*d*) *Single Limit With Preference.*—If the requirements demand a limit in only one direction and there is a preferred value, the preferred value should be indicated together with the maximum or minimum value.

Example:

Weight of coating (to the nearest 0.1 mg. per sq. in.)..... { 10.0 mg. per sq. in., preferred / 2.0 mg. per sq. in., min. }

(*e*) *Single Limit Without Preference.*—If the requirements demand a limit in only one direction and there is no preference for a specific value, a maximum or minimum value alone should be indicated.

Example:

Weight of coating (to the nearest 0.1 mg. per sq. in.)..... { 2.0 mg. per sq. in., min. }

(*f*) *Percentage Maximum Permissible Deviations.*—If the permissible plus or minus deviation is a direct function of the preferred value, it may be expressed as a percentage, particularly if a wide range of preferred values is expressed or accepted by custom.

Examples:

The twist, in turns per inch to the nearest turn, shall be as specified, plus or minus 7 per cent.

The resistance in ohms per foot at 20 C. shall be as specified, plus or minus 10 per cent; to the nearest 0.001 ohm for gages Nos. 14 to 21, to the nearest 0.01 ohm for gages Nos. 22 to 31, and to the nearest 0.1 ohm for gages Nos. 32 to 40. On the basis of this specification, the table of minimum and maximum values to be used for purposes of acceptance and rejection would be as follows:

Wire Gage	Resistance,[a] ohm per foot at 20 C.		
	Min.	Specified	Max.
No. 14	0.145	0.161	0.177
No. 15	0.183	0.203	0.223
.....	.....	.....	.....
.....	.....	.....	.....
.....	.....	.....	.....
No. 21	0.732	0.813	0.894
No. 22	0.93	1.03	1.13
.....	.....	.....	.....
.....	.....	.....	.....
.....	.....	.....	.....
No. 31	7.50	8.33	9.16
No. 32	9.3	10.3	11.3
.....	.....	.....	.....
.....	.....	.....	.....
.....	.....	.....	.....
No. 39	48.7	54.1	59.5
No. 40	56.0	73.3	80.6

[a] The following applies to all specified limits in this table: For purposes of acceptance and rejection, an observed value or a calculated value obtained by test should be rounded off "to the nearest unit" in the last right-hand place of figures used in expressing the limiting value.

Averages

9. (*a*) **Where the term "average" is used without modifying adjectives, it shall be interpreted to indicate the arithmetic mean.** If some other average is intended, such as the weighted arithmetic mean, the geometric mean, etc., the standard should so state.

(*b*) Standards should indicate clearly whether specified limits are to be applied to each single reading, to the average of all readings, to the average of certain subgroups of readings, etc.

(*c*) If the places of figures to be considered significant in an average differ from those expressed in the specified limit for a single specimen, a statement should be made to this effect.

Nonnumerical Terms

10. (*a*) The expressions in the following Paragraphs (*b*) to (*d*) are not numerical in character but have a definite relationship to numerical requirements in standards.

(*b*) *Remainder, Balance, or Difference.*—The analytical method should, in general, include the determination of all the essential ingredients and all of the impurities for which numerical requirements are specified. The term "remainder" (rather than "balance" or "difference") should be used when it is not necessary to make a direct determination of the remaining constituent or constituents involved.

	Chemical Composition, per cent (to the nearest 0.1 per cent)
Example:	
Copper	4.5 ± 0.5
Iron	1.0 max.
Silicon	2.5 ± 0.5
Other constituents (magnesium + zinc + manganese)	0.5 max.
Aluminum	remainder

(*c*) *Trace, None, Nil.*—The use of indefinite terms, such as "trace," "none," "nil" should be avoided unless defined as "none by such and such a test."

(*d*) *Total Impurities.*—The use of indefinite expressions, such as "total impurities," "total other impurities," etc., should be avoided unless the specification likewise lists the elements that should be considered under such expressions.

Test Method

11. (*a*) The standard should indicate clearly the test or analytical method to be followed for determining each of the specified numerical requirements. This may be done as follows:

(*1*) By reference to an A.S.T.M. standard,

(*2*) By describing the method in detail, or

(*3*) By stating "any approved method."

(*b*) Procedures (*1*) and (*2*) are recommended as preferable to (*3*).

Permissible Variations for Controlled Constants of Test

12. Permissible variations for controlled constants of testing apparatus and equipment should be designated.

Example:

Thickness test— "..... with a load of 0.100 ± 0.001 psi. distributed over a circular area of 3.00 ± 0.01 in. in diameter."

Tensile strength test— ".....with a jaw speed of 12.00 ± 0.50 in. per min."

In the above examples the underlining designates places of figures to be considered significant for purposes of acceptance and rejection *of conditions of test.*

Permissible Variations for Condition of Test Specimens

13. The physical condition of the specimen at the time of test should be specified in all details which influence the characteristics being tested.

Examples:

Molded Insulating Materials, Physical Tests.—All specimens shall be conditioned before testing by drying in an oven at 50 ± 5 C. (122 ± 9 F.) for 48 hr., followed by cooling to room temperature in a desiccator.

Woven Textile Fabrics, Physical Tests.—Standard condition of woven fabrics shall be that reached by the fabric when in moisture equilibrium with a standard atmosphere having a relative humidity of 65 per cent at 70 F. (21 C.). A deviation of plus or minus 2.0 per cent is permitted in relative humidity and plus or minus 2 F. (1.1 C.) in temperature.

Sampling and Acceptance Requirements

14. (*a*) If it is the intent of the specification to provide a definite basis for acceptance and rejection with respect to the numerical requirements, the sampling procedure should be outlined in detail, including:

(*1*) Method of selecting samples,

(*2*) Number of tests or analyses, and

(*3*) Acceptance requirements—including (*i*) conditions to be met by results of tests if lot is to be considered acceptable, and (*ii*) conditions for and methods of resampling and retesting.

(*b*) If there are restrictions as to the number of specimens, tests, or readings to be permitted in obtaining an average for purposes of acceptance and rejection, the standard should so state.

Test Readings

15. (*a*) Where it is important in any operation of a test or an analysis, the specification should indicate how many places of figures the tester or analyst should read on the scale of measurement.

Example:

Measure to the nearest 0.001 in.
Read to the nearest 10 lb.
Weigh to the nearest 0.01 g.
Read to the limit of sensitivity of the .....

(*b*) If the direct reading of a test is taken in the same units of measurement as designated in the specified limit, and if the phrase "to the nearest....." (or underlining) is used in designating the specified limit, this method of expression will indicate to how many places of figures it is *necessary* to read purely for purposes of acceptance and rejection. The practice of reading and recording *additional* places of figures is recommended; among other things, this practice makes available information that may be useful in studies or investigations as to variability of material or of testing methods.

Example:

Specified Limit: Weight
(to the nearest 0.1 g.).....5.0 g., min.

For purposes of acceptance and rejection, it would be *necessary* to read the scales of the bal-

ance only to the nearest 0.1 g. If, however, it were subsequently desirable to review past data to study variability, and if the range of variation were only say one or two tenths of a gram, the amount of information available would be meager if values had been recorded only to the nearest tenth of a gram, but would be plentiful if values had been recorded to the nearest hundredth of a gram.

Calculations

16. (*a*) The basis of all percentages should be given.

(*b*) It is recommended that all calculations, such as determinations of tensile strength in pounds per square inch, corrections for temperatures, etc., should carry at least two more places of figures than are to be considered significant for purposes of acceptance and rejection.

Example.—The specified tensile strength of a material is 50,000 psi., min. (to the nearest 100 psi.), that is, three places of figures are to be considered significant for purposes of acceptance and rejection. This recommendation calls for carrying out calculations to five places of figures.

If maximum load readings for two test specimens are 5340 and 5080 lb., and if the calculated cross-sectional areas are 0.10240 and 0.10174 sq. in., respectively, the calculated values of the tensile strength would be as follows:

$$\frac{5340}{0.10240} = 52{,}148 \text{ psi.}$$

$$\frac{5080}{0.10174} = 49{,}931 \text{ psi.}$$

Note that if the recommendation in Paragraph (*b*) had not been followed and if only *one* more place of figures (than are to be considered significant for acceptance and rejection) had been used, the second calculated value would have been 5080/0.1017 = 49,951 psi. This value would then have been rounded off to 50,000 and would have been considered *acceptable,* whereas it should have been considered *rejectable.* Using "two more places" in calculations, in accordance with the practice here recommended, usually prevents this type of calculation error from affecting the final value used for purposes of acceptance and rejection.

(*c*) Digits that are not to be considered in acceptance values should be rounded off (see Section 4 (*b*)) after all calculations, including averaging, are completed.

Example.—Calculated values of Section 16 (*b*) would be rounded off to give final values of 52,100 and 49,900 psi., respectively, for purposes of acceptance and rejection.

Tentative Recommended Practice for

CHARACTERISTICS OF STANDARD CARBON ARC ACCELERATED WEATHERING UNIT[1]

A.S.T.M. Designation: E 42 – 42 T

ISSUED, 1942.[2]

This Tentative Recommended Practice of the American Society for Testing Materials is under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. This recommended practice covers the basic principles of an accelerated weathering unit of the carbon arc type.

NOTE.—Inasmuch as weather varies from day to day and place to place, thus making it impossible to correlate with the weather at all times and for all locations, it is desirable to promote the adoption of such cycles as will reproduce the particular weathering influences that are of interest. It is believed that the characteristics recommended herein cover those points of fundamental importance that will provide an acceptable accelerated weathering unit for many purposes, and at the same time permit the development of detailed and specific cycles needed for the obtaining of particular data and falling within the general structure of this recommended practice. As an example, if the emphasis is on rust-inhibiting paint, in addition to any cycle adopted, it may be desirable to have an accessory high humidity cabinet made available.

Apparatus

2. (*a*) The light source shall be a carbon arc.

(*b*) The carbons shall be of such composition and operating under such conditions that the quality of the spectrum as it strikes the test specimens shall approach so far as possible the spectrum of the sun.

(*c*) The weathering unit shall include means for measuring and controlling the following:

(*1*) Current,
(*2*) Voltage,
(*3*) Temperature of air and water, and
(*4*) Running time.

(*d*) The materials of construction shall be of such character as not to react with the test specimens during the test.

Procedure

3. (*a*) Light values shall be measured by the oxalic acid - uranyl sulfate method described in the Tentative Method of Calibrating a Light Source Used for Accelerating the Deterioration of Rubber (A.S.T.M. Designation: D 749).[3]

(*b*) Specimens shall be mounted vertically and shall rotate about the arc in order to provide uniform distribution of the light. If the specimens are mounted

[1] Under the standardization procedure of the Society, this recommended practice is under the jurisdiction of the A.S.T.M. Committee E-1 on Methods of Testing.

[2] Accepted by Committee E-10 on Standards, August 24, 1942.

[3] 1944 Book of A.S.T.M. Standards, Part III.

vertically both above and below the horizontal center line of the light source, their position should be transposed periodically to provide uniform distribution of the light in a vertical plane over the entire face of the specimen. Products of combustion shall not be permitted to touch the specimens.

(*c*) The air temperature at the distance of the surface of the specimens under test shall be constant. It shall be determined by a shielded thermometer at a point most remote from the water spray.

(*d*) Water sprays shall be provided whereby clean water such as drinking water may be forced on the specimens to simulate the washing action of rain, to provide moisture for causing alternate expansion and contraction due to swelling and drying out, and to introduce thermal shock and sweating. No recirculation or immersion in the spray water shall be permitted.

NOTE.—Certain types of water may contain constituents deleterious to the materials under test.

(*e*) The accelerated weathering unit at all times shall be operated under constant voltage at the recommended arc amperage. The proper carbons shall be used, the globes and filters cleaned at frequent intervals, and the temperatures of the air and water shall be regulated within the tolerances established.

TENTATIVE DEFINITIONS WITH PROCEDURES RELATING TO CONDITIONING AND WEATHERING[1]

A.S.T.M. Designation: E 41 - 42 T

ISSUED, 1942.[2]

This Tentative Standard of the American Society for Testing Materials is, under its Regulations, subject to annual revision. Suggestions for revision should be addressed to the Society, 260 S. Broad St., Philadelphia 2, Pa.

1. These definitions and test procedures pertain to the conditioning and weathering of materials for test purposes. Unless otherwise specified, they are intended to apply to all cases where combinations of atmospheric influences are an essential part of materials testing.

Definitions

2. (*a*) *Conditioning.*—The process of subjecting a material to a stipulated influence or combination of influences for a stipulated period of time. The influences may be relative humidity, temperature, light, pressure, air composition, or other factors, used jointly or severally.

NOTE 1.—In some cases a material is subjected to a stipulated influence or combination of influences until substantial equilibrium is reached. Because of the labor involved in determining when equilibrium has been reached, it is customary to subject the material to the stipulated influences for a stipulated period of time, the period of time having been determined with due regard to the nature of the material, the atmospheric influences, and the conditioning equipment.

NOTE 2.—Conditioning may be used for (*a*) predicting service behavior, (*b*) obtaining reproducible results, (*c*) testing for acceptance, (*d*) research, and (*e*) process control; for any single objective or combination of objectives.

NOTE 3.—It is generally true that only a portion of the influences enumerated in the above definition are applicable to any given material. Hence, in any discussion of conditioning, the particular influences that are significant must be stated.

(*b*) *Average Room Conditions.*— 40 per cent relative humidity at a temperature of 77 F. (25 C.).

(*c*) *Dry Room Conditions.*—15 per cent relative humidity at a temperature of 85 F. (29 C.).

(*d*) *Moist Room Conditions.*— 75 per cent relative humidity at a temperature of 77 F. (25 C.).

(*e*) *Absolute Humidity.*[3,4]—Concentration (density) of water vapor in the atmosphere expressed as weight per unit volume or weight per unit weight

[1] Under the standardization procedure of the Society, these definitions are under the jurisdiction of the A.S.T.M. Committee E-1 on Methods of Testing.

[2] Accepted by Committee E-10 on Standards, August 24, 1942.

[3] These definitions are identical with those appearing in the Standard Method of Determining Relative Humidity (A.S.T.M. Designation: D 337), 1944 Book of A.S.T.M. Standards, Part III.

[4] Editorially revised in 1944.

of (*1*) dry air or (*2*) water vapor and air. Grams per cubic foot, grains per pound, or pounds per pound are commonly used.

(*f*) *Relative Humidity.*[3,4]—Ratio of actual pressure of existing water vapor to maximum possible pressure of water vapor in the atmosphere at the same temperature, expressed as a percentage.

(*g*) *Dew Point.*[3]—Temperature at which condensations of moisture would occur if the existing atmosphere were cooled without change in vapor pressure.

(*h*) *Vapor Pressure.*[3]—Component of atmospheric pressure caused by presence of water vapor, usually expressed in inches of mercury.

(*i*) *Dry Bulb Temperature.*[3]—Temperature of the air as indicated by a standard thermometer.

(*j*) *Wet Bulb Temperature.*[3]—For purposes of this method, temperature indicated by the wet bulb thermometer of a standard sling psychrometer or its equivalent. Theoretically, temperature at which the atmosphere would become saturated by evaporation of water without loss or gain in total heat content of the air and vapor.

(*k*) *Hygrometer.*[3]—Any properly calibrated instrument which indicates directly or indirectly the humidity of the air.

(*l*) *Absorption Hygrometer.*[3]—Any one of several types of hygrometers containing a hygroscopic substance the length, thickness, or weight of which is a measurable index of the humidity of the atmosphere. Usually such devices are graduated directly either in relative humidity or in regain of stock in process, and are continuously indicating.

(*m*) *Wet and Dry Bulb Hygrometer or Psychrometer.*[3]—Contains two matched thermometers, one plain and one having a wet bulb covering as hereinafter specified—for simultaneously indicating the dry and wet bulb temperatures of the atmosphere.

(*n*) *Recording Hygrometer or Psychrometer.*[3]—Any approved type of hygrometer or psychrometer equipped with automatic clocking and recording device capable of making a continuous time - relative humidity autograph, or time - wet and dry bulb temperature autograph.

(*o*) *Sling Psychrometer.*[3]—A psychrometer containing matched dry and wet bulb thermometers suitably mounted for swinging through the atmosphere, for simultaneously indicating dry and wet bulb temperatures. The thermometers shall be so mounted as to act independently and to face the air current during swinging.

Relative Humidity Determination

3. (*a*) Relative humidity shall be determined in accordance with the Standard Method of Determining Relative Humidity (A.S.T.M. Designation: D 337) of the American Society for Testing Materials.[5]

(*b*) For determining relative humidity from psychrometric observations within the normal temperature range 0 to 112 F. (−20 to +45 C.), reference may for convenience be made to the Psychrometric Table for Relative Humidity.[6]

Room Temperature Determination

4. Whenever the room temperature is to be determined in any test method the following procedures shall be used:

(*a*) All air temperatures shall be measured with an A.S.T.M. Gravity Thermometer E 1 (12F - 39) or E 1 (12C - 39) of the total immersion type, having a range of −5 to +215 F. or −20 to +102 C., as specified, and conforming to the requirements of the

[5] 1944 Book of A.S.T.M. Standards, Part III.

[6] *Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part I, p. 567 (1931); also in "A.S.T.M. Standards on Textile Materials," October, 1944, p. 379, available as a separate publication.

Standard Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1) of the American Society for Testing Materials.[7]

(*b*) All air temperature (room temperature) measurements shall be made with the thermometer located within 2 ft. in a horizontal direction of the point at which the test is actually being made. In case the test involves any heat or heating apparatus, the thermometer shall be shielded from direct radiation as well as convection currents.

[7] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Standard Laboratory Atmosphere

5. Whenever the materials to be tested are known to be sensitive to variations in temperature or moisture, or both, the tests shall be conducted in a room or chamber of controlled humidity and temperature. Unless otherwise specified, the tests shall be made in the standard laboratory atmosphere having a relative humidity of 50 ± 2 per cent at a temperature of 77 ± 2 F. (25 ± 1.1 C.).

TENTATIVE REVISIONS

OF

A.S.T.M. STANDARDS ON NONMETALLIC MATERIALS—CONSTRUCTIONAL

As of November, 1944

(For change of status in any of these tentative revisions, see supplementary publications to be issued in 1945 and 1946.)

Tentative revisions of A.S.T.M. Standards are printed for one or more years with a view of eliciting criticisms, of which the committee concerned will take due cognizance before recommending final action.

New Tentative Revisions and any changes in these Tentative Revisions, issued in 1945 and 1946, will appear in the 1945 and 1946 Supplements to the Book of A.S.T.M. Standards. It is important that these Supplements be consulted in case any change of status has occurred with respect to any of these Tentative Revisions.

Members of the Society and others are invited to direct written criticisms of any of these Tentative Revisions to the Headquarters of the Society, 260 S. Broad St., Philadelphia 2, Pa.

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[1] See Supplements to be issued in 1945 and 1946 for revision or change of status of any of these tentative revisions.

TENTATIVE REVISIONS OF A.S.T.M. STANDARDS RELATING TO NONMETALLIC MATERIALS—CONSTRUCTIONAL

As of November, 1944

Standard Specifications for Masonry Cement (C 91 – 40):[1]

The Tentative Specifications for Masonry Cement (C 91 – 44 T)[2] are intended to replace, when adopted, the present Standard Specifications C 91 – 40.

Tentative revision, submitted August, 1944.

Standard Methods of Chemical Analysis of Portland Cement (C 114 – 44):[3]

The Tentative Methods of Chemical Analysis of Portland Cement (C 114 – 44 T)[4] are intended to be added, when adopted, to the present Standard Methods C 114 – 44.

Tentative revision, submitted June, 1944.

Standard Definitions of Terms Relating to Lime (C 51 – 44):[5]

The Tentative Definitions of Terms Relating to Lime (C 51 – 44 T)[6] are intended to be added, when adopted, to the present Standard Definitions C 51 – 44.

Tentative revision, submitted June, 1944.

Standard Specifications for Calcined Gypsum for Dental Plasters (C 72 – 40):[7]

Section 8.—Change to read as follows by the omission of the words in brackets:

8. The chemical analysis and physical properties of calcined gypsum for use in the preparation of dental plasters shall be determined in accordance with the Standard Methods of Testing Gypsum and Gypsum Products (A.S.T.M. Designation: C 26).[8] [Except that in the determination of the time of setting and the tensile strength the calcined gypsum and water shall be mixed in the proportion of 100 g. of calcined gypsum to 60 ml. of water. The calcined gypsum shall be allowed to soak in distilled water at 70 F. (21 C.) for 30 sec., and shall then be stirred for 30 sec. and placed immediately in the molds.]

Tentative revision, submitted June, 1942.

Standard Methods of Testing Gypsum and Gypsum Products (C 26 – 42):[8]

Section 19 (b).—Add a note to read as follows:

NOTE.—The use of 0.1 per cent retarder in determining the testing consistency is necessary when working with quick-setting calcined gypsum products.

Tentative revision, submitted June, 1942.

Standard Definitions of Terms Relating to Gypsum (C 11 – 41):[9]

The Tentative Definitions of Terms Relating to Gypsum (C 11 – 41 T)[10] are intended to be added, when adopted, to the present Standard Definitions C 11 – 41.

Tentative revision, submitted June, 1941.

Standard Specifications for Ground Fire Clay as a Mortar for Laying-Up Fire-clay Brick (C 105 – 41):[11]

New Section.—Add the following new Section 7, renumbering the present Section 7 as Section 8:

7. *Retests.*—Because of variables resulting from sampling and the lack of satisfactory reproducibility in tests conducted by different laboratories, the material may be resampled and retested when requested by either the purchaser or the manufacturer. This may apply in instances when the first test results do not conform to the requirements prescribed in these specifications.

[1] See p. 7.
[2] See p. 1206.
[3] See p. 14.
[4] See p. 1214.
[5] See p. 121.
[6] See p. 1243.
[7] See p. 133.
[8] See p. 144.
[9] See p. 158.
[10] See p. 1244.
[11] See p. 267.

The final result to be used shall be the average of at least two sets of results, each of which has been obtained by following in detail the specified testing procedures.

Tentative revision, submitted June, 1944.

Standard Method of Test for Pyrometric Cone Equivalent (P.C.E.) of Refractory Materials (C 24 – 42):[12]

Section 3.—Change from its present form: namely,

3. (*a*) The dried sample shall be thoroughly mixed and after the addition of sufficient dextrine, glue, gum tragacanth, or other alkali-free organic binder and water, shall be formed in a truncated trigonal pyramid with its base at a small angle to the trigonal axis, and of the following dimensions: Over-all height (between planes perpendicular to the trigonal axis) $1\frac{1}{8}$ in.; length of side of the truncated tip, $\frac{1}{32}$ in.; length of the two longer sides of the base, $\frac{3}{32}$ in.; base at an external angle of 82 deg. to one face and perpendicular to the symmetry plane through the opposite edge of the pyramid.

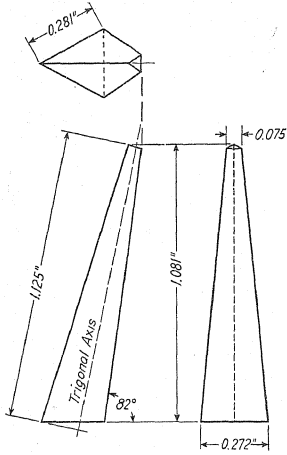


FIG. 1.—Standard Pyrometric Test Cone.

(*b*) When dry the test cones may be subjected, when necessary, to a preliminary burn at a temperature not exceeding 2370 F. (1300 C.) for the purpose of sintering them to a firm condition to permit handling.

to read as follows:

3. (*a*) Samples of unfired clays (Note), or of mixes containing appreciable proportions of raw clay, shall, after being prepared in accordance with Section 2 (*a*), be heated in an oxidizing atmosphere in the temperature range of 1700 to 1800 F. (925 to 980 C.) for not less than 30 min.

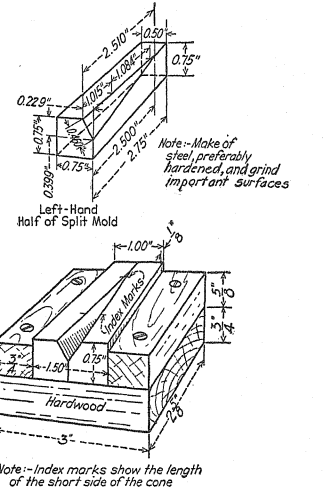


FIG. 2.—Split Mold for A.S.T.M. Pyrometric Test Cone.

NOTE.—Some unfired clays bloat when they are formed into cones and are carried through the high-temperature heat treatment prescribed in Section 5 (*a*) without preliminary calcining. The substances that cause bloating can, in most cases, be expelled by heating the clay samples before testing.

(*b*) The clay sample may be given the heat treatment prescribed in Paragraph (*a*) after it has been formed into a cone (Paragraph (*c*)), but this procedure has been found not as effective as the treatment of the powdered material. Should

[12] See p. 294.

cones so prepared bloat during the P.C.E. test, a portion of the original sample shall be heated in its powdered condition as prescribed in Paragraph (*a*) and then retested.

(*c*) The dried sample shall be thoroughly mixed and after the addition of sufficient dextrine, glue, gum tragacanth, or other alkali-free organic binder and water, shall be formed in a metal mold into test cones in the shape of a truncated trigonal pyramid with its base at a small angle to the trigonal axis, and of the dimensions shown in Fig. 1. The mold to be used in forming the test cone is shown in Fig. 2.

Tentative revision, submitted March, 1943; Modified, June, 1944

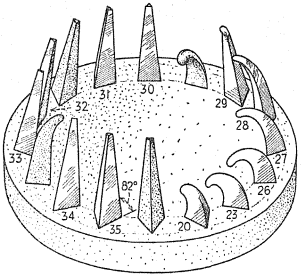


FIG. 3.—Method of Mounting Test Cones and Appearance After Testing.

Section 4.—Change to read as follows by the addition of the italicized word and figure and the omission of those in brackets:

4. The test cones shall be mounted on plaques of refractory material of such a composition as will not affect the fusibility of the cones. They shall be mounted with the base embedded approximately 3 mm. (0.12 in.) in the plaque and the face of one side inclined at an angle of 82 deg. with the horizontal. The arrangement of the test cones with respect to the Standard Pyrometric Cones shall be substantially as shown in [Fig. 1] *Fig. 3*, that is, alternating the test cones with the Standard Pyrometric Cones in so far as is practical (Note). The plaque may be any convenient size and shape and may be biscuited before using, if desired.

Tentative revision, submitted March, 1943.

Standard Method of Panel Test for Resistance to Thermal and Structural Spalling of Refractory Brick (C 38 – 42):[13]

Section 4.—Change to read as follows by the addition of the italicized figures and the omission of those in brackets:

4. The specimens shall be laid up so as to form a test panel not less than 18 in. square, so that a 9 by 2½-in. surface of each specimen will be exposed to the heat treatment. Each specimen shall be labeled with ceramic paint on the face opposite that to be tested, and then weighed to the nearest [0.1] *0.05* lb. There shall be placed about the test panel a suitable grade of refractory dummy brick, in the manner shown. All brick in the test panel shall be laid up with refractory kaolin unless otherwise specified. The joints shall be not thicker than $\frac{1}{16}$ in. The test panel, including dummy brick, shall be insulated in accordance with the arrangement and design shown.

Tentative revision, submitted March, 1943.

Section 5.—Change to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

5. The prepared test panels shall be placed in position on either side of the preheating furnace. When only one lot of brick is to be tested, a dummy panel should be used. The joint between the ends of the panel dummy brick and the preheating furnace shall be sealed with plastic kaolin. During the heating of the panels, the surface of the test specimen opposite that being heated shall be insulated in the specified manner so as to maintain a suitable temperature gradient in the specimen. The furnace shall be so operated that the temperature specified for preheating shall be reached in not less than 5 nor more than 8 hr. and maintained 24 hr. The permissible variation in temperature may be [plus 35 F. and minus 45 F. (plus 20 C. and minus 25 C.)] *plus or minus 20 F. (10 C.)*, but the average of the temperature during the run shall be that specified. After completing the preheating, the test panel shall be left in

[13] See p. 297.

position for not less than 8 hr. before being removed. As a result of this heat treatment, any tendency toward fusion, vitrification, swelling, or shrinkage will be evident and any development of structural spalling will be apparent. A record shall be made of the condition of the brick specimens, indicating the extent of the fusion, vitrification, swelling, or shrinkage. If shrinkage takes place a record shall be made of its nature, that is, whether or not this occurs as a shrinkage of each brick as a unit, or as the development of cracks on the surface of each brick. The surface of the test panels may be photographed.

Tentative revision, submitted March, 1943.

Section 7.—Change to read as follows by the addition of the italicized figures and the omission of the figures and words in brackets:

7. When the test panel has cooled sufficiently so that the brick can be handled, the panel shall be carefully dismantled. Each of the test specimens shall be cleaned to free it of kaolin and in such a manner as to remove spalls, after which the specimens shall be reweighed to the nearest [0.1] *0.05* lb., and the loss in weight recorded as percentage of the original weight. [When a second heating treatment is given and the 9-in. brick specimens have been numbered from 1 to 7 from top to bottom in the test panel, their order shall be changed so as to assume the positions of 4–3–2–1–7–6–5. In the case of the six 3-in. brick specimens the order shall be changed so as to assume the positions of 3–2–1–6–5–4 for the second heating treatment. The specimens shall also be turned end for end.]

Tentative revision, submitted March, 1943.

Standard Method of Panel Test for Resistance to Thermal and Structural Spalling of High Heat Duty Fireclay Brick (C 107 – 42):[14]

Section 4 (d).—Change to read as follows by the addition of the italicized figures and the omission of the figures and words in brackets; also by omission of the Note following this section:

(*d*) The furnace gas pressure during the 24-hr. preheating period shall be within the limits of [0.3 to 0.6] *0.4 to 0.5* in. of water [for gas-fired furnaces].

[Note.—In furnaces used for the purpose of obtaining control and experimental data where the method is followed in all details except that oil is used as a fuel, it is recommended that the furnace gas pressure be maintained between 0.1 and 0.2 in. of water.]

Tentative revision, submitted March, 1943.

Standard Method of Panel Test for Resistance to Thermal and Structural Spalling of Super Duty Fireclay Brick (C 122 – 42):[15]

Section 4 (d).—Change to read as follows by the addition of the italicized figures and the omission of the figures and words in brackets; also by omission of the Note following this section:

(*d*) The furnace gas pressure during the 24-hr. preheating period shall be within the limits of [0.3 to 0.6] *0.4 to 0.5* in. of water [for gas-fired furnaces].

[Note.—In furnaces used for the purpose of obtaining control and experimental data where the method is followed in all details except that oil is used as a fuel, it is recommended that the furnace gas pressure be maintained between 0.1 and 0.2 in. of water.]

Tentative revision, submitted March, 1943.

Standard Definitions of Terms Relating to Refractories (C 71 – 42):[16]

Under the section on properties, add the definition of a new term to read as follows:

Calcining of Refractory Materials.—The heat treatment to which raw refractory materials are subjected, preparatory to further processing or use, for the purpose of eliminating volatile chemically combined constituents and producing volume changes.

Tentative revision, submitted June, 1935; Modified, June, 1942; March, 1943.

Standard Definition of the Term Sand (C 58 – 28):[17]

The Tentative Definition of the Term Aggregate (C 58 – 28 T)[18] is intended to be added, when adopted, to the present Standard Definition C 58 – 28.

Tentative revision, submitted June, 1928.

[14] See p. 304.

[15] See p. 306.
[16] See p. 330.
[17] See p. 452.
[18] See p. 1339.

Standard Specifications for Preformed Expansion Joint Fillers for Concrete (Nonextruding and Resilient Types) (D 544 – 41):[19]

Section 1.—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

1. These specifications cover preformed expansion joint fillers of the following [four] *five* types for use in concrete construction. The type or types desired shall be specified by the purchaser.

Type I, cork,
Type II, self-expanding cork,
Type III, sponge rubber, [and]
Type IV, cork rubber, *and*
Type V, bituminous fiber.

Tentative revision, submitted November, 1943.

Section 2.—Add a new Paragraph (*d*) to read as follows:

(*d*) *Bituminous Fiber.*—This type shall consist of preformed strips which have been formed from cane or other suitable fibers of a cellular nature, securely bound together and uniformly impregnated with a suitable bituminous binder.

Tentative revision, submitted November, 1943.

Section 4.—Change to read as follows by the addition of the italicized words and figures:

4. (*a*) Recovery.—The test specimen shall be given three applications of a load sufficient to compress the material to 50 per cent of its thickness before test. The load shall be immediately released after each application. At the end of 1 hr. after the third application, the joint shall have recovered to at least 90 per cent of its thickness before test *for types I, II, III, and IV, and to at least 70 per cent of its thickness before test for type V.*

(*b*) Compression.—The load required to compress the test specimen to 50 per cent of its thickness before test shall not be less than 100 nor more than 750 psi. *For bituminous fiber joint filler (type V), the material after compression shall not show a loss of more than 3 per cent of its original weight.*

(*c*) Extrusion.—The test specimen shall be compressed to 50 per cent of its thickness before test with three of the edges restrained. The amount of extrusion of the free edge shall not exceed 0.25 in.

(*d*) Expansion.—In the case of self-expanding cork (type II) expansion joint filler only, the test specimen after being immersed in boiling water for 1 hr. shall have a final thickness not less than 140 per cent of the thickness before test. Discoloration of the water shall not be considered an indication of failure.

(*e*) Boiling in Hydrochloric Acid.—In the case of cork and self-expanding cork (types I and II) expansion joint fillers only, the test specimen when boiled for 1 hr. in hydrochloric acid (sp. gr. 1.19) shall show no evidence of disintegration. Discoloration, or a small amount of swelling shall not be considered as failure.

(*f*) Weathering Test.—A weathering test may be conducted in accordance with the procedure specified in Section 8. Test specimens shall show no evidence of disintegration when subjected to the specified weathering test. *Delamination or separation of the fiber structure in type V shall be considered disintegration.* Test specimens which have been subjected to the weathering test may be required to meet the requirements for recovery, compression, and extrusion specified in Paragraphs (*a*), (*b*), and (*c*).

(*g*) *Penetration of Recovered Bitumen.—The penetration at 25 C. of the bitumen recovered from the bituminous fiber joint filler (type V) shall be between 25 and 100.*

(*h*) *Bitumen Content.—The bitumen content of the bituminous fiber filler (type V) shall be at least 35 per cent by weight.*

Tentative revision, submitted November, 1943.

Standard Methods of Testing Preformed Expansion Joint Fillers for Concrete (Nonextruding and Resilient Types) (D 545 – 41):[20]

Section 1.—Change to read as follows by the addition of the italicized words:

1. These methods cover the following tests for preformed expansion joint fillers for concrete: expansion in boiling water, recovery, compression, extrusion, boiling in hydrochloric acid, weathering, *penetration of recovered bitumen, and bitumen content.* The recovery, compression, and extrusion tests shall be performed at a laboratory room temperature of 70 F. (21 C.) or above.

Tentative revision, submitted November, 1943.

[19] See p. 509.

[20] See p. 518.

Section 2 (*a*).—Change to read as follows by the addition of the italicized words:

2. (*a*) In the case of cork, sponge rubber, cork rubber, *and bituminous fiber* (types I, III, IV, *and V*) expansion joint fillers only, five specimens for test shall be cut from each sample. Each test specimen shall be freshly and squarely cut using a metal plate 4 by 4 in. as a cutting template. The metal template shall be machined from ¼-in. plate to fit the extrusion mold described in Section 5 (*b*). The template shall fit the mold within minus 0.005 in. in length and minus 0.005 in. in width.

Tentative revision, submitted November, 1943.

Section 4 (*a*).—Change to read as follows by the addition of the italicized words:

4. Recovery and Compression, *and Loss of Bitumen During Compression*: (*a*) Test Specimens.—One of the test specimens prepared as described in Section 2 (*a*) (or one of the expanded specimens prepared as described in Section 2 (*b*) in the case of self-expanding cork, type II) shall be used for these tests. In the case of cork, sponge rubber, cork rubber, *and bituminous fiber* (types I, III, IV, *and V*) expansion joint fillers, these tests shall be made on specimens of the material as received. However, if the cork filler (type I) fails to meet the requirements specified, check tests shall be made on specimens which have been immersed in water for 24 hr. and subsequently air dried for 24 hr. Acceptance shall be based on the results of these check tests.

Tentative revision, submitted November, 1943.

Section 5 (*a*).—Change to read as follows by the addition of the italicized words:

5. (*a*) Test Specimens.—One of the test specimens prepared as described in Section 2 (*a*) (or one of the expanded specimens prepared as described in Section 2 (*b*) in the case of self-expanding cork, type II) shall be used for this test. In the case of cork, sponge rubber, cork rubber, *and bituminous fiber* (types I, III, IV, *and V*) expansion joint fillers, these tests shall be made on specimens of the material as received. However, if the cork filler (type I) fails to meet the requirements of the specifications, check tests shall be made on specimens which have been immersed in water for 24 hr. and subsequently air dried for 24 hr. Acceptance shall be based on the results of these check tests.

Tentative revision, submitted November, 1943.

New Sections.—Add new Sections 8 and 9 on penetration of recovered bitumen and bitumen content to read as follows:

8. *Penetration of Recovered Bitumen.*—(*a*) A 150 to 200-g. sample of the joint filler shall be accurately weighed to the nearest 0.1 g., broken up, and placed in a centrifugal extractor.[21] Pure benzene shall then be added in sufficient quantity to saturate the sample which shall then be allowed to soak for 30 min. to dissolve the bitumen. The bitumen-benzene solution shall be extracted by centrifuging the sample. Several successive washes of pure benzene shall be added to the centrifuge bowl until the extracted solvent is not discolored, and all the bitumen saturant has been removed from the fiber joint filler.

(*b*) The bitumen-benzene solution and the washings shall be collected in a 2-l. round-bottom, heat-resistant glass flask,[22] and distilled over an oil bath with a water-cooled condenser for condensing the benzene vapors. The distillation shall be continued until the bitumen-benzene solution is concentrated to approximately 200 ml. The distillation shall be stopped at this point, the residue poured into a 300-ml. distillation flask, and distilled to 360 C. in accordance with the Standard Method of Test for Distillation of Cut-Back Asphaltic Products (A.S.T.M. Designation: D 402).[23]

(*c*) The penetration under 100-g. load for 5 sec. at 25 C. (77 F.) of the recovered bitumen from the distillation described in Paragraph (*b*) shall be determined in accordance with the Standard Method of Test for Penetration of Bituminous Materials (A.S.T.M. Designation: D 5).[24]

9. *Bitumen Content.*—The fiber residue after the extraction of the bitumen in accordance with Section 8 (*a*) shall be dried to constant weight and the bitumen content determined by difference, expressed as a percentage by weight of the original sample. At the option of the purchaser, the bitumen content shall be determined on samples after they have been heated in a constant-temperature oven at 163 C. for a period of 3 hr.

Tentative revision, submitted November, 1943.

Standard Methods of Chemical Analysis of Calcium Chloride (D 345 – 34):[25]

Title.—Change to read as follows by

[21] A Rotarex type extractor is suitable for this purpose.
[22] Pyrex glass is satisfactory for this purpose.
[23] See p. 552.
[24] See p. 573.
[25] See p. 522.

the addition of the italicized words and the omission of those in brackets:

Standard Method[s] of [Chemical Analysis] *Sampling and Testing* Calcium Chloride

Tentative revision, submitted August, 1941.

Section 1.—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

1. These methods cover procedures for the *sampling* and [chemical analysis] *testing* of calcium chloride.

Tentative revision, submitted August, 1941.

New Sections.—Add the following new Sections 2 and 3, renumbering the subsequent sections accordingly:

2. *Sampling.*—A mesh lot of calcium chloride shall be sampled for testing purposes as follows:

(*a*) Not less than three containers shall be selected at random from the lot. Each of the containers shall be sampled by scraping aside the top layer to a depth of approximately 1 in. and taking 1-lb. samples by means of a sampling thief or other method which will insure a sample that is representative of a cross-section of the material in the container to a depth of at least 6 in. Precautions should be taken during the sampling operation to avoid exposing the sample unduly to atmospheric moisture. The individual sample shall be immediately and thoroughly mixed to form a representative composite sample of material, and stored in a sealed glass container.

3. *Sieve Analysis.*—Approximately 200 g. of calcium chloride weighed to the nearest 0.1 g. shall be sieved in accordance with the Standard Method of Test for Sieve Analysis of Fine and Coarse Aggregates (A.S.T.M. Designation: C 136),[25] utilizing the ¾-in., ½-in., and No. 20 sieves together with a bottom pan and a cover (sieving shall be completed within a period of approximately 1 min.). The sieve analysis shall be reported on the basis of the grading requirements specified in Section 3 of the Standard Specifications for Calcium Chloride (A.S.T.M. Designation: D 98).[27]

Tentative revision, submitted August, 1941.

[25] See p. 441.
[27] See p. 511.

Standard Method of Mechanical Analysis of Soils (D 422 – 39):[28]

Section 6 (*b*).—Change to read as follows by the addition of the italicized words:

(*b*) At the conclusion of this shaking the time shall be recorded, the graduate placed in the bath, and readings taken with the hydrometer at the end of both 1 and 2 min. The hydrometer shall be read at the top of the meniscus formed by the suspension around its stem. If hydrometer A *or the streamlined hydrometer graduated in grams of soil per liter of suspension* is used, it shall be read to the nearest 0.5 g. per liter. Hydrometer B shall be read to the nearest 0.0005 specific gravity. Subsequent readings shall be taken at intervals of 5, 15, 30, 60, 250, and 1440 min. after the beginning of sedimentation. Readings on the thermometer placed in the constant temperature bath shall be made coincidentally with the hydrometer readings and recorded.

Tentative revision, submitted August, 1943.

Section 14.—Add the following to this section as an alternative report:

Alternative Report.—The results of complete mechanical analyses furnished by the combined sieve and hydrometer analyses shall be reported as follows:

Sieve Analysis

Sieve Size	Percentage Passing
2-in.	
1½-in.	
1-in.	
¾-in.	
⅜-in.	
No. 4 (4760-micron)	
No. 10 (2000-micron)	
No. 40 (420-micron)	
No. 60 (250-micron)	
No. 200 (74-micron)	

Hydrometer Analysis

Size of Particle	Percentage Smaller Than
0.05 mm	
0.005 mm	
0.001 mm	

For materials examined for any particular type of work or purpose, only such fractions shall be reported as are included in the specifications or other requirements for the work or purpose.

Tentative revision, submitted August, 1942.

[28] See p. 616.

Standard Specifications for Zinc Sulfide Pigments (D 477 – 41):[29]

Table I.—Make the following changes in the respective requirements for composition:

Zinc Sulfide-Barium Pigment:
Change minimum barium sulfate requirement from "98" to "95" per cent of remainder.
Zinc Sulfide-Magnesium Pigment:
Change minimum magnesium and micaceous silicates requirement from "98" to "95" per cent of remainder.
Lithopone:
Change minimum barium sulfate requirement from "98" to "95" per cent of remainder.
Titanated Lithopone:
Change minimum barium sulfate requirement from "98" to "95" per cent of remainder.

Tentative revision, submitted August, 1944.

Standard Methods of Sampling and Analysis of Shellac (D 29 – 40):[30]

The Tentative Method of Test for Color of Orange Shellac (D 29 – 41 T)[31] is intended to be added, when adopted, to the present Standard Methods D 29 – 40.

Tentative revision, submitted June, 1941.

Standard Methods of Sampling and Testing Lacquer Solvents and Diluents (D 268 – 44):[32]

The Tentative Method of Test for Water in Lacquer Solvents and Diluents (D 268 – 44 T)[33] is intended to be added, when adopted, to the present Standard Methods D 268 – 44.

Tentative revision, submitted June, 1944.

Standard Specifications for A.S.T.M. Thermometers (E 1 – 44):[34]

The Tentative Specifications for A.S.T.M. Thermometers (E 1 – 44 T)[35] are intended to be added, when adopted, to the present Standard Specifications E 1 – 44.

Tentative revision, submitted June, 1944.

[29] See p. 828.
[30] See p. 993.
[31] See p. 1538.
[32] See p. 1053.
[33] See p. 1535.
[34] See p. 1129.
[35] See p. 1567.

LIST OF A.S.T.M. STANDING COMMITTEES

(November, 1944)

A. Ferrous Metals

Committee:

A-1 on Steel.
A-2 on Wrought Iron.
A-3 on Cast Iron.
A-4 (*Discontinued*)
A-5 on Corrosion of Iron and Steel.
A-6 on Magnetic Properties.
A-7 on Malleable-Iron Castings.
A-8 (*Merged with Committee A-6*)
A-9 on Ferro-Alloys.
A-10 on Iron-Chromium, Iron-Chromium-Nickel, and Related Alloys.
Advisory Committee on Corrosion.

B. Non-Ferrous Metals

Committee:

Coordinating Committee on Non-Ferrous Metals and Alloys.
B-1 on Copper and Copper-Alloy Wires for Electrical Conductors.
B-2 on Non-Ferrous Metals and Alloys.
B-3 on Corrosion of Non-Ferrous Metals and Alloys.
B-4 on Electrical-Heating, Electrical-Resistance, and Electric-Furnace Alloys.
B-5 on Copper and Copper Alloys, Cast and Wrought.
B-6 and Die-Cast Metals and Alloys.
B-7 on Light Metals and Alloys, Cast and Wrought.
B-8 on Electrodeposited Metallic Coatings.
B-9 on Metal Powders and Metal Powder Products.

C. Cementitious, Ceramic, Concrete, and Masonry Materials

Committee:

C-1 on Cement.
C-2 (*Discontinued*)
C-3 (*Merged with Committee C-15*)
C-4 on Clay Pipe.
C-5 on Fire Tests of Materials and Construction.
C-6 on Drain Tile.
C-7 on Lime.
C-8 on Refractories.
C-9 on Concrete and Concrete Aggregates.
C-10 (*Merged with Committee C-15*)
C-11 on Gypsum.
C-12 on Mortars for Unit Masonry.
C-13 on Concrete Pipe.
C-14 on Glass and Glass Products.
C-15 on Manufactured Masonry Units.
C-16 on Thermal Insulating Materials.
C-18 on Natural Building Stones.

D. Miscellaneous Materials

Committee:

D-1 on Paint, Varnish, Lacquer, and Related Products.
D-2 on Petroleum Products and Lubricants.
D-3 on Gaseous Fuels.
D-4 on Road and Paving Materials.
D-5 on Coal and Coke.
D-6 on Paper and Paper Products.
D-7 on Wood.
D-8 on Bituminous Waterproofing and Roofing Materials.
D-9 on Electrical Insulating Materials.
D-10 on Shipping Containers.
D-11 on Rubber and Rubber-Like Materials.
D-12 on Soaps and Other Detergents.
D-13 on Textile Materials.
D-14 on Adhesives.
D-15 (*Reconstituted as Technical Committee XII of Committee E-1*)
D-16 on Industrial Aromatic Hydrocarbons
D-17 on Naval Stores.
D-18 on Soils for Engineering Purposes.
D-19 on Water for Industrial Uses.
D-20 on Plastics.

E. Miscellaneous Subjects

Committee:

E-1 on Methods of Testing.
E-2 on Spectrographic Analysis.
E-3 on Chemical Analysis of Metals.
E-4 on Metallography.
E-5 on Standing Committees.
E-6 on Papers and Publications.
E-7 on Radiographic Testing.
E-8 on Nomenclature and Definitions.
E-9 on Research.
E-10 on Standards.

INDEX

BOOK OF A.S.T.M. STANDARDS, PART II

STANDARDS AND TENTATIVE STANDARDS ON NONMETALLIC MATERIALS—CONSTRUCTIONAL

This Index covers solely those standards appearing in this volume. A combined Index, covering all standards and tentative standards appearing in all three Parts of the Book of Standards, is furnished separately. The Combined Index will be revised annually to incorporate the latest references to the standards whether they appear in the Book of Standards or in the 1945 and 1946 Supplements thereto. The Combined Index will be particularly helpful after one of the Supplements has been issued.

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